

(FILE 'HOME' ENTERED AT 10:40:00 ON 18 SEP 2008)

FILE 'REGISTRY' ENTERED AT 10:40:21 ON 18 SEP 2008

L1 80 S (2-7)/LI AND P AND 3.5-8/O

FILE 'CAPLUS' ENTERED AT 10:40:52 ON 18 SEP 2008

L2 59 S L1

L3 8 S L2 AND (TI OR V OR CR OR MN)

L4 11 S L2 AND (FE OR CO OR NI OR CU OR ZR)

L5 6 S L2 AND (NB OR MO OR RU OR AG OR TA OR W OR PT OR AU)

=> s l3 or l4 or l5

L6 18 L3 OR L4 OR L5

=> d 1-18 ibib ti it abs

L6 ANSWER 1 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2006:496431 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 145:145864

TITLE: A bimetallic, coordinated-ketene complex formed from a bimetallic lithium-carbon spirocycle by lithium-mediated insertion of CO into a rhodium-carbon bond

AUTHOR(S): Fang, Min; Jones, Nathan D.; Lukowski, Robert; Tjathas, Jim; Ferguson, Michael J.; Cavell, Ronald G.

CORPORATE SOURCE: Department of Chemistry, University of Alberta, Edmonton, AB, T6G 2G2, Can.

SOURCE: Angewandte Chemie, International Edition (2006), 45(19), 3097-3101

CODEN: ACIEF5; ISSN: 1433-7851

PUBLISHER: Wiley-VCH Verlag GmbH & Co. KGaA

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 145:145864

TI A bimetallic, coordinated-ketene complex formed from a bimetallic lithium-carbon spirocycle by lithium-mediated insertion of CO into a rhodium-carbon bond

IT Insertion reaction

(bimetallic, ketene complex formed from bimetallic lithium-carbon spirocycle by Li-mediated insertion of CO into rhodium-carbon bond and crystal structures of reactant and product)

IT Carbene complexes

Spiro compounds

RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(bimetallic, ketene complex formed from bimetallic lithium-carbon spirocycle by Li-mediated insertion of CO into rhodium-carbon bond and crystal structures of reactant and product)

IT Alkenes, preparation

RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(heterocumulenes, ketenes, Li-Rh tetranuclear complex; bimetallic, ketene complex formed from bimetallic lithium-carbon spirocycle by Li-mediated insertion of CO into rhodium-carbon bond and crystal structures of reactant and product)

IT Crystal structure

Molecular structure

(of lithium rhodium diene iminophosphoranylmethylene dinuclear and carbonyl ketene tetranuclear complexes)

IT 12092-47-6, Bis[chloro(1,5-cyclooctadiene)rhodium] 230969-40-1

RL: RCT (Reactant); RACT (Reactant or reagent)
 (bimetallic, ketene complex formed from bimetallic lithium-carbon
 spirocycle by Li-mediated insertion of CO into rhodium-carbon
 bond and crystal structures of reactant and product)

IT 898550-45-3P
 RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP
 (Preparation); RACT (Reactant or reagent)
 (crystal structure; bimetallic, ketene complex formed from bimetallic
 lithium-carbon spirocycle by Li-mediated insertion of CO into
 rhodium-carbon bond and crystal structures of reactant and product)

IT 898550-48-6P
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
 (crystal structure; bimetallic, ketene complex formed from bimetallic
 lithium-carbon spirocycle by Li-mediated insertion of CO into
 rhodium-carbon bond and crystal structures of reactant and product)

IT 898550-47-5P
 RL: SPN (Synthetic preparation); PREP (Preparation)
 (hydrolysis product; bimetallic, ketene complex formed from bimetallic
 lithium-carbon spirocycle by Li-mediated insertion of CO into
 rhodium-carbon bond and crystal structures of reactant and product)

IT 898550-46-4P
 RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP
 (Preparation); RACT (Reactant or reagent)
 (mol. structure, hydrolysis; bimetallic, ketene complex formed from
 bimetallic lithium-carbon spirocycle by Li-mediated insertion of
 CO into rhodium-carbon bond and crystal structures of reactant
 and product)

AB A bimetallic spirocyclic bridged-carbene complex (1; carbene =
 (TMSN:PPh₂)₂C) was prepared and reacts almost quant. with CO by
 formal insertion into the Rh-C(Li) bond to form the dimeric tetranuclear
 complex 2, the 1st example of a $\mu_2, \eta^2-(O,C)$ Li-Rh ketene complex.
 Complex 2 reacts with H₂O to transform the ketene ligand into a methanide
 group by CO elimination. The crystal and mol. structures of 1
 and 2·Et₂O were determined by x-ray crystallog.

REFERENCE COUNT: 60 THERE ARE 60 CITED REFERENCES AVAILABLE FOR THIS
 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 2 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2006:330088 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 145:46176

TITLE: Coordination chemistry of isomeric mixtures of linked
 di(phosphaguanidine) compounds: a spectroscopic and
 crystallographic study

AUTHOR(S): Mansfield, Natalie E.; Coles, Martyn P.; Avent,
 Anthony G.; Hitchcock, Peter B.

CORPORATE SOURCE: Department of Chemistry, University of Sussex,
 Falmer/Brighton, BN1 9QJ, UK

SOURCE: Organometallics (2006), 25(10), 2470-2474
 CODEN: ORGND7; ISSN: 0276-7333

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 145:46176

TI Coordination chemistry of isomeric mixtures of linked di(phosphaguanidine)
 compounds: a spectroscopic and crystallographic study

IT Phosphines
 RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP
 (Preparation); RACT (Reactant or reagent)
 (di-; preparation and reactions of diastereomeric mixts. of rac- and
 meso-di(phosphaguanidine) compds. with aluminum and platinum to give
 bimetallic aluminum and monometallic platinum complexes)

IT Crystal structure
Molecular structure
(of diastereomeric di(phosphaguanidine) ligands and their bimetallic aluminum and monometallic platinum complexes)

IT 890054-94-1P 890055-36-4P
RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(crystal structure; preparation and reactions of diastereomeric mixts. of rac- and meso-di(phosphaguanidine) compds. with aluminum and platinum to give bimetallic aluminum and monometallic platinum complexes)

IT 889470-57-9P 889470-58-0P 889470-60-4P 889470-61-5P
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(crystal structure; preparation and reactions of diastereomeric mixts. of rac- and meso-di(phosphaguanidine) compds. with aluminum and platinum to give bimetallic aluminum and monometallic platinum complexes)

IT 75-24-1, Trimethylaluminum 538-75-0 554-68-7, Triethylammonium chloride 693-13-0 12080-32-9, Dichloro(1,5-cyclooctadiene)platinum 12266-72-7, (1,5-Cyclooctadiene)diodoplatinum 125083-71-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation and reactions of diastereomeric mixts. of rac- and meso-di(phosphaguanidine) compds. with aluminum and platinum to give bimetallic aluminum and monometallic platinum complexes)

IT 889470-62-6P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and reactions of diastereomeric mixts. of rac- and meso-di(phosphaguanidine) compds. with aluminum and platinum to give bimetallic aluminum and monometallic platinum complexes)

IT 889470-59-1P 889470-63-7P 890055-48-8P 890055-50-2P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and reactions of diastereomeric mixts. of rac- and meso-di(phosphaguanidine) compds. with aluminum and platinum to give bimetallic aluminum and monometallic platinum complexes)

AB Multinuclear NMR spectroscopy and x-ray diffraction techniques were used to identify diastereomeric mixts. of rac- and meso-di(phosphaguanidine) compds. A brief survey of their coordination chemical demonstrated the formation of N,N'-bound bimetallic Al species and monometallic Pt complexes in which the ligand chelates to the metal through the two P donor atoms.

REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 3 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 2003:573904 CAPLUS <<LOGINID::20080918>>
DOCUMENT NUMBER: 140:62169
TITLE: Electrical conductivity in Li-Si-P-O-N oxynitride thin-films
AUTHOR(S): Lee, Seung-Joo; Bae, Jun-Hyun; Lee, Hee-Won; Baik, Hong-Koo; Lee, Sung-Man
CORPORATE SOURCE: Department of Metallurgical Engineering, Yonsei University, Seoul, 120-749, S. Korea
SOURCE: Journal of Power Sources (2003), 123(1), 61-64
CODEN: JPSODZ; ISSN: 0378-7753
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
TI Electrical conductivity in Li-Si-P-O-N oxynitride thin-films
IT Battery electrolytes
(elec. conductivity of Li-Si-P-O-N thin-film electrolytes for lithium batteries)
IT 639079-90-6 639079-91-7, Lithium nitride oxide phosphide

silicate (Li_{1.9}NO_{0.26}P(SiO₃)_{0.28}) 639079-92-8, Lithium nitride
oxide phosphide silicate (Li_{2.9}N_{1.26}O_{0.1}P(SiO₄)_{0.35}) 639079-93-9,
Lithium nitride oxide phosphide silicate (Li_{2.9}N_{1.3}O_{0.25}P(SiO₃)_{0.45})
RL: DEV (Device component use); PRP (Properties); USES (Uses)
(elec. conductivity of Li-Si-P-O-N thin-film electrolytes for lithium
batteries)

AB N-containing Li silicophosphate (LiSiPON) thin-film electrolytes, which
contain 2 glass-forming elements, are fabricated by sputtering from a
(1-x)Li₃PO₄·xLi₂SiO₃ target in a N reactive plasma. The results of
impedance measurements show that the activation energy for conduction
decreases as the Si content increases, which increases the ionic conductivity
of
the films. These improvements in the elec. properties of the films are
due to the combined effect of the mixed former and N incorporation. The
decomposition potential of the electrolyte film in contact with Pt is
.apprx.5.5 V.

REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 4 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2003:364301 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 139:214572

TITLE: Reaction of a cyclopentadienylcobalt(I) phosphane
chelate with trimethylsilyl chloride and some
pseudohalides: unanticipated formation of new
cyclopentadienylcobalt(II) and (III) chelates

AUTHOR(S): Kakoschke, Alf; Yong, Li; Wartchow, Rudolf;
Butenschoen, Holger

CORPORATE SOURCE: Institut fuer Organische Chemie, Universitaet
Hannover, Hannover, D-30167, Germany

SOURCE: Journal of Organometallic Chemistry (2003), 674(1-2),
86-95

CODEN: JORCAI; ISSN: 0022-328X

PUBLISHER: Elsevier Science B.V.

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 139:214572

TI Reaction of a cyclopentadienylcobalt(I) phosphane chelate with
trimethylsilyl chloride and some pseudohalides: unanticipated formation of
new cyclopentadienylcobalt(II) and (III) chelates

IT Transition metal halides

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(cobalt, pseudohalides, crystal structure; preparation of
cyclopentadienylcobalt(II) (tethered phosphine) chloride and
pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT Sandwich compounds

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(half-sandwich, cobalt, tethered phosphine; preparation of
cyclopentadienylcobalt(II) (tethered phosphine) chloride and
pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT Oxidation

(halogenation; preparation of cyclopentadienylcobalt(II) (tethered
phosphine) chloride and pseudohalide complexes by trimethylhalosilane-
mediated halogenation)

IT Crystal structure

Molecular structure

(of cyclopentadienyl cobalt(II)-tethered phosphine chloride, isocyanate
and azide complexes)

IT Addition reaction, coordinative

(oxidative; of trimethylsilyl cyanide to give cobalt(III) dicyano
cyclopentadienyl (tethered phosphine) complex)

IT Halogenation
(preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 21502-53-4, Lithium diisopropylphosphide
RL: RCT (Reactant); RACT (Reactant or reagent)
(addition, oxidation; preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 176683-07-1P 588729-48-0P 588729-49-1P 588729-52-6P
588729-53-7P
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(crystal structure; preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 588729-51-5P
RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(mol. structure, lithiation, oxidation; preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 7677-24-9, Trimethylsilyl cyanide
RL: RGT (Reagent); RACT (Reactant or reagent)
(oxidative addition; preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 1118-02-1, Trimethylsilyl isocyanate 2290-65-5, Trimethylsilyl isothiocyante
RL: RGT (Reagent); RACT (Reactant or reagent)
(oxidation reagent; preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 141847-75-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(oxidation, halogenation; preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 1450-14-2P, Hexamethyldisilane
RL: BYP (Byproduct); PREP (Preparation)
(preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

IT 588729-50-4P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of cyclopentadienylcobalt(II) (tethered phosphine) chloride and pseudohalide complexes by trimethylhalosilane-mediated halogenation)

AB Reactions of the (η^2 -ethene)[$(\eta^5:\eta^1$ -2-[(di-tert-butyl)phosphino]ethyl)cyclopentadienyl- κ P]cobalt(I) chelate complex 1 with trimethylsilyl chloride and some pseudohalides were examined. Reaction of 1 with trimethylsilyl chloride, azide, isocyanate, and isothiocyante gave cobalt(II) chelates [$(\eta^5:\eta^1)$ -C₅H₄CH₂CH₂PtBu₂- κ P]CoX] (2-5, X = Cl, N₃, NCO, NCS), some of which were characterized by x-ray structure anal. In contrast to these reactions, treatment of 1 with trimethylsilyl cyanide resulted in the formation of cobalt(III) chelate [$(\eta^5:\eta^1)$ -C₅H₄CH₂CH₂PtBu₂- κ P]Co(CN)₂] (7). Reaction of 7 with LiPr₂ followed by oxidation gave a small yield of tetrametallic complex [$(\eta^5$ -tBuPOCH₂CH₂C₅H₄)Co(CN)(μ -iPr₂POLi- κ P)]₂ (8), in which the two cobalt centers are bridged by an (LiO)₂ rhombus.

REFERENCE COUNT: 59 THERE ARE 59 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 5 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2002:894424 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 138:106770

TITLE: [Li{CH(Me)P(Ph)₂(NCO₂Me)}₂(THF)₂]: Crystal, Solution, and Calculated Structure of a N-Delocalized Lithium Phosphazene

AUTHOR(S): Fernandez, Ignacio; Alvarez Gutierrez, Julia Maria; Kocher, Nikolaus; Leusser, Dirk; Stalke, Dietmar; Gonzalez, Javier; Ortiz, Fernando Lopez

CORPORATE SOURCE: Area de Quimica Organica, Universidad de Almeria Carretera de Sacramento s/n, Almeria, 04120, Spain

SOURCE: Journal of the American Chemical Society (2002), 124(51), 15184-15185

CODEN: JACSAT; ISSN: 0002-7863

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 138:106770

TI [Li{CH(Me)P(Ph)₂(NCO₂Me)}₂(THF)₂]: Crystal, Solution, and Calculated Structure of a N-Delocalized Lithium Phosphazene

IT Equilibrium
(monomer-dimer; of alkyl di-Ph methoxycarbonyl phosphazene lithium complex)

IT Crystal structure
Molecular structure
(of alkyl di-Ph methoxycarbonyl phosphazene lithium complex)

IT MP2 (second-order Moller-Plesset method)
(of delocalized lithium phosphazene complex)

IT Molecular structure
(optimized; of delocalized lithium phosphazene complex)

IT 488082-70-8 488082-71-9
RL: CPS (Chemical process); PEP (Physical, engineering or chemical process); PRP (Properties); PROC (Process)

(preparation, crystal, solution, and calculated structure of a N-delocalized lithium phosphazene)

IT 488082-73-1P
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(preparation, crystal, solution, and calculated structure of a N-delocalized lithium phosphazene)

IT 127802-82-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation, crystal, solution, and calculated structure of a N-delocalized lithium phosphazene)

AB The first crystal structure of the lithium complex of a P-alkyl-P,P-diphenyl(N-methoxycarbonyl)phosphazene, [Li{CHMePPh₂:NCO₂Me}]₂(THF)₂, prepared by treatment of CH₃CH₂P(Ph)₂NCO₂Me with BuLi in THF at -30° for 30 min and then layering the orange solution with hexane at -18° for 2 days, is described. It is dimeric, with the anion chelating the lithium in an unusual six-membered ring. A monomer-dimer equilibrium has been identified in THF solution Ab initio calcs.

indicated that the six-membered ring is electronically favored over an alternative Li-C-P-N four-membered ring.

REFERENCE COUNT: 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 6 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2002:84941 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 136:279555

TITLE: Water-Promoted Reaction of a Platinum(II) Oxo Complex with Ethylene

AUTHOR(S): Flint, Bruce; Li, Jian-Jun; Sharp, Paul R.
 CORPORATE SOURCE: Department of Chemistry, University of
 Missouri-Columbia, Columbia, MO, 65211, USA
 SOURCE: Organometallics (2002), 21(5), 997-1000
 CODEN: ORGND7; ISSN: 0276-7333
 PUBLISHER: American Chemical Society
 DOCUMENT TYPE: Journal
 LANGUAGE: English
 OTHER SOURCE(S): CASREACT 136:279555

TI Water-Promoted Reaction of a Platinum(II) Oxo Complex with Ethylene
 IT Rearrangement catalysts
 (formation of platinum phosphine hydroxy complex as catalyst for oxygen
 atom transfer reaction in reaction of platinum oxo complexes with
 ethylene)
 IT Crystal structure
 Molecular structure
 (of platinum lithium phosphine hydroxy bridged complex)
 IT Substitution reaction, coordinative
 (of platinum oxo complexes with ethylene, during which oxygen atom
 transfer takes place)
 IT Rearrangement
 (oxygen atom transfer; in reaction of platinum oxo complexes with
 ethylene, catalyzed by platinum phosphine hydroxy complex)
 IT 406462-95-1P
 RL: CAT (Catalyst use); SPN (Synthetic preparation); PREP (Preparation);
 USES (Uses)
 (preparation and catalyst for oxygen atom transfer)
 IT 406462-91-7P
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
 (Reactant or reagent)
 (preparation and substitution reaction with dppe)
 IT 406462-94-0P
 RL: CAT (Catalyst use); PRP (Properties); SPN (Synthetic preparation);
 PREP (Preparation); USES (Uses)
 (preparation of)
 IT 406462-96-2P 406462-97-3P
 RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation of)
 IT 406462-93-9P
 RL: CAT (Catalyst use); PRP (Properties); SPN (Synthetic preparation);
 PREP (Preparation); USES (Uses)
 (preparation, crystal structure, and attempted catalyst for oxygen atom
 transfer)
 IT 74-85-1, Ethylene, reactions 7732-18-5, Water, reactions 172800-85-0
 406462-90-6 406462-98-4
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (water-promoted reaction of a platinum oxo complex with ethylene)
 AB Treatment of [(dppp)Pt(μ -O)]₂(LiOTf)₂ (dppp = Ph₂P(CH₂)₃PPh₂)
 with ethylene in the presence of trace amts. of water results in oxygen
 atom transfer to one arm of the bidentate phosphine ligand and formation
 of (dpppO)Pt(η ²-CH₂:CH₂)₂ (dpppO = Ph₂P(CH₂)₃P(O)Ph₂).
 Further investigation reveals that the reaction of [L₂Pt(μ -O)]₂(LiOTf)₂
 (L = dppp, dppb) with water forms (L)₂Pt(OH)₂, which acts as a catalyst
 for the oxygen atom transfer reaction. The analogous oxo complex
 [(PPh₃)₂Pt(μ -O)]₂(LiBF₄)₂ does not react with ethylene under similar
 conditions. These results indicate that hydroxo complex intermediates
 should be considered in oxygen atom transfer reactions.

REFERENCE COUNT: 42 THERE ARE 42 CITED REFERENCES AVAILABLE FOR THIS
 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

ACCESSION NUMBER: 1998:625562 CAPLUS <<LOGINID::20080918>>
 DOCUMENT NUMBER: 129:339029
 ORIGINAL REFERENCE NO.: 129:68921a,68924a
 TITLE: Phosphorus oxonitridosodalites: synthesis using a molecular precursor and structural investigation by x-ray and neutron powder diffraction and ³¹P MAS NMR spectroscopy
 AUTHOR(S): Stock, Norbert; Irran, Elisabeth; Schnick, Wolfgang
 CORPORATE SOURCE: Laboratorium Anorganische Chemie Universitat, Bayreuth, D-95440, Germany
 SOURCE: Chemistry--A European Journal (1998), 4(9), 1822-1828
 CODEN: CEUJED; ISSN: 0947-6539
 PUBLISHER: Wiley-VCH Verlag GmbH
 DOCUMENT TYPE: Journal
 LANGUAGE: English

TI Phosphorus oxonitridosodalites: synthesis using a molecular precursor and structural investigation by x-ray and neutron powder diffraction and ³¹P MAS NMR spectroscopy
 IT Crystal structure
 Molecular structure
 (of copper and lithium phosphorus oxonitridosodalites)
 IT 13597-72-3, Phosphoric triamide
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for preparation of copper and lithium phosphorus oxonitridosodalites)
 IT 7664-41-7, Ammonia, reactions 7783-20-2, Ammonium sulfate ((NH₄)₂SO₄), reactions 10026-13-8, Phosphorus chloride (PCl₅)
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for preparation of copper and lithium phosphorus oxonitridosodalites using a mol. precursor)
 IT 13966-08-0P 182505-10-8P
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
 (for preparation of copper and lithium phosphorus oxonitridosodalites using a mol. precursor)
 IT 7758-89-6, Copper chloride (CuCl) 22722-08-3, Phosphenimidic nitride
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for preparation of copper phosphorus oxonitridosodalite)
 IT 10377-51-2, Lithium iodide (LiI) 12124-97-9, Ammonium bromide 12136-58-2, Lithium sulfide (Li₂S) 13455-05-5, Phosphorothioic triamide
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for preparation of lithium phosphorus oxonitridosodalite)
 IT 7447-41-8, Lithium chloride, reactions
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (for preparation of lithium phosphorus oxonitridosodalite using a mol. precursor)
 IT 215191-22-3P 215191-26-7P 215191-27-8P 215191-28-9P
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
 (preparation and crystal structure)
 AB The oxonitridophosphates M₈-mHm[P₁₂N₁₈O₆]Cl₂ (M = Cu, Li) with a sodalite-like [P₁₂N₁₈O₆]⁶⁻ framework of corner-sharing PN₃O tetrahedra were synthesized by the reaction of the resp. metal chlorides with (NH₂)₂P(O)NP(NH₂)₃·NH₄Cl. In this precursor the desired molar ratio, P:O = 2:1, of the [P₁₂N₁₈O₆]⁶⁻ framework structure was preorganized on a mol. level. Analogous oxonitridosodalites also were obtained from the metal salts MX (M = Cu, Li; X = Cl, Br, I) or Li₂S, the P/O and P/N components OP(NH₂)₃ or HPN₂, and NH₄X or MX as halogen sources. The crystal structures of the phosphorus oxonitridosodalites Cu₄.8H₃.2[P₁₂N₁₈O₆]Cl₂ (1), Li₅.5H₂.5[P₁₂N₁₈O₆]Cl₂ (2), Li₆.2H₁.8[P₁₂N₁₈O₆]Br₂ (3), and Li₅.8H₂.2[P₁₂N₁₈O₆]I₂ (4) were studied by using neutron and x-ray powder diffraction as well as ³¹P MAS NMR

spectroscopy. Rietveld refinements were performed in the cubic space group I.hivin.43m ($Z = 1$, $a = 820.25(1)$ to $830.81(2)$ pm for $X = Cl$ to I). No exptl. evidence for a crystallog. ordering of the N/O atoms and for other than PN_3O tetrahedra in the sodalite frameworks was obtained.

REFERENCE COUNT: 35 THERE ARE 35 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 8 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1996:279252 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 125:24809

ORIGINAL REFERENCE NO.: 125:4671a, 4674a

TITLE: Co-ordination chemistry of tridentate phosphinothiolates; syntheses and structures of $[Li_4\{PhP(C_6H_3S-2-SiMe_3-3)_2\}_2(MeOCH_2CH_2OMe)_2]$, $[Sn_2\{PhP(C_6H_3S-2-SiMe_3-3)_2\}_2]$ and $[NHet_3][Sm\{PhP(C_6H_3S-2-R-3)_2\}_2(py)_2] \cdot 2py$ ($R = H$ or $SiMe_3$, $py = C_5H_5N$)

AUTHOR(S): Froelish, Nikolaus; Hitchcock, Peter B.; Hu, Jin; Lappert, Michael F.; Dilworth, Jonathan R.

CORPORATE SOURCE: Sch. Chem. Molecular Sciences, Univ. Sussex, Brighton, BN1 9QJ, UK

SOURCE: Journal of the Chemical Society, Dalton Transactions: Inorganic Chemistry (1996), (9), 1941-1946
CODEN: JCDBTBI; ISSN: 0300-9246

PUBLISHER: Royal Society of Chemistry

DOCUMENT TYPE: Journal

LANGUAGE: English

TI Co-ordination chemistry of tridentate phosphinothiolates; syntheses and structures of $[Li_4\{PhP(C_6H_3S-2-SiMe_3-3)_2\}_2(MeOCH_2CH_2OMe)_2]$, $[Sn_2\{PhP(C_6H_3S-2-SiMe_3-3)_2\}_2]$ and $[NHet_3][Sm\{PhP(C_6H_3S-2-R-3)_2\}_2(py)_2] \cdot 2py$ ($R = H$ or $SiMe_3$, $py = C_5H_5N$)

IT Crystal structure
Molecular structure
(of lithium and tin and samarium phenylbis(mercaptophenyl)phosphine complexes)

IT 119327-23-0

RL: RCT (Reactant); RACT (Reactant or reagent)
(for preparation of lithium and tin and samarium phenylbis(mercaptophenyl)phosphine complexes)

IT 177594-36-4, Triiodobis(tetrahydrofuran)samarium

RL: RCT (Reactant); RACT (Reactant or reagent)
(for preparation of samarium phenylbis(mercaptophenyl)phosphine complexes)

IT 119327-18-3

RL: RCT (Reactant); RACT (Reactant or reagent)
(for preparation of samarium phenylbis(mercaptophenyl)phosphine pyridine complex)

IT 177594-29-5DP, reaction products with ytterbium iodide

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(preparation and NMR of)

IT 177594-29-5P 177594-31-9P 177594-33-1P 177594-35-3P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(preparation and crystal structure of)

IT 19357-86-9DP, Ytterbium diiodide, reaction product with lithium phenylbis(mercaptophenyl)phosphine complex

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

AB Treatment of $PhP[C_6H_3(SH-2)R-3]_2$ ($R = H$ (H_2L_1) or $SiMe_3$ (H_2L_2)) with SmI_3 and Net_3 and then recrystn. from pyridine (py) yielded $[NHet_3][SmL_2(py)_2] \cdot 2py$ ($L = L_1$ (3) or L_2 (4)), while H_2L_2 and $LiBu$ or $[Sn\{N(SiMe_3)_2\}_2]$ afforded $[Li_2L_2(dme)]_2$ (1) (after addition of dme) or $[(SnL_2)_2]$ (2), resp. ($dme = MeOCH_2CH_2OMe$). 1-4 Were characterized by

microanal., NMR spectra and single-crystal x-ray diffraction.

L6 ANSWER 9 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1994:218017 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 120:218017

ORIGINAL REFERENCE NO.: 120:38729a,38732a

TITLE: Chiral tripod ligand: one pot reaction $\text{MeC}(\text{CH}_2\text{PPh}_2)_3 \rightarrow$

$\text{MeC}(\text{CH}_2\text{P}(\text{Ph})(\text{R}))_3$; intermediate, diastereoselective control and complex chemistry

AUTHOR(S): Walter, Olaf; Klein, Thomas; Huttner, Gottfried;

Zsolnai, Laszlo

CORPORATE SOURCE: Anorganisch-Chemisches Institut der Universitaet Heidelberg, Im Neuenheimer Feld 270, Heidelberg, 6900, Germany

SOURCE: Journal of Organometallic Chemistry (1993), 458(1-2), 63-81

CODEN: JORCAI; ISSN: 0022-328X

DOCUMENT TYPE: Journal

LANGUAGE: German

OTHER SOURCE(S): CASREACT 120:218017

TI Chiral tripod ligand: one pot reaction $\text{MeC}(\text{CH}_2\text{PPh}_2)_3 \rightarrow$
 $\text{MeC}(\text{CH}_2\text{P}(\text{Ph})(\text{R}))_3$; intermediate, diastereoselective control and complex chemistry

IT Crystal structure

Molecular structure

(of chiral tripod phosphorus ligand molybdenum complexes)

IT 22031-12-5

RL: RCT (Reactant); RACT (Reactant or reagent)

(lithiation and sequential alkylation or molybdenum complexation of)

IT 79190-90-2P 153956-06-0P 153956-43-5P 154006-92-5P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(preparation and crystal structure of)

IT 153956-39-9P 153956-40-2P 154006-91-4P 154096-26-1P

RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and multinuclear NMR of)

IT 153956-41-3P 153956-42-4P 154006-93-6P

RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

IT 145011-41-2P 145033-21-2P 153888-54-1P 153888-55-2P 153888-56-3P

153888-57-4P 154002-80-9P 154002-81-0P 154002-82-1P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation, NMR, and complexation of, with molybdenum)

IT 153956-44-6P 154005-24-0P

RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation, mol. structure, or alkylation of)

IT 15038-48-9

RL: RCT (Reactant); RACT (Reactant or reagent)

(reaction of, with chiral tripod phosphorus ligand)

AB The tripod ligand $\text{H}_3\text{CC}(\text{CH}_2\text{PPh}_2)_3$, 1, reacts with lithium metal to produce $\text{H}_3\text{CC}(\text{CH}_2\text{PPhLi})_3$, 2, in good yields. Compound 2 crystallizes in the form of $2 \cdot 4\text{THF} \cdot \text{MeN}(\text{CH}_2\text{CH}_2\text{NMe}_2)_2$. The crystal structure of this adduct shows bridging as well as terminal phosphorous-coordination of lithium. Compound 2 reacts with electrophiles RX to give $\text{H}_3\text{CC}(\text{CH}_2\text{P}(\text{Ph})(\text{R}))_3$, 3 (R = H, Me, Et, CHMe₂, CH₂Ph, 3a-3e). The transformation $1 \rightarrow 2 \rightarrow 3$ can be performed in a one-pot reaction. While the chiral compds. 3 may be characterized as such, their reaction with $(\text{CH}_3\text{CN})_3\text{Mo}(\text{CO})_3$ yields the easy-to-characterize coordination compds. $\text{H}_3\text{CC}(\text{CH}_2\text{P}(\text{Ph})(\text{R}))_3\text{Mo}(\text{CO})_3$, 5. NMR anal. of 3 and 5 shows that from the two diastereomeric forms of 3 (the enantiomeric pair SSR/RRS and the pair SSS/RRR) only the SSR/RRS-diastereomer is formed with R = CH₂Ph (3e, 5e). In the other

cases (R = H, Me, Et, CHMe2) a statistical 3 : 1 mixture of the two diastereomers is formed. These findings are further corroborated by x-ray analyses of H3CC(CH2P(Ph)(Et))3Mo(CO)3, 5c, and H3CC(CH2P(Ph)(CH2Ph))3Mo(CO)3, 5e. The remarkable facts reported in this paper are the ease with which the chiral tripod ligands H3CC(CH2P(Ph)(R))3, 3, are prepared, and the evidence that their formation may well involve diastereoselective control.

L6 ANSWER 10 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1993:449648 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 119:49648

ORIGINAL REFERENCE NO.: 119:9021a,9024a

TITLE: Contributions to the chemistry of boron. 214.
Synthesis and reaction chemistry of
aminophosphinoboranes

AUTHOR(S): Dou, Danan; Westerhausen, Matthias; Wood, Gary L.;
Linti, Gerald; Duesler, Eileen N.; Noth, Heinrich;
Paine, Robert T.

CORPORATE SOURCE: Dep. Chem., Univ. New Mexico, Albuquerque, NM, 87131,
USA

SOURCE: Chemische Berichte (1993), 126(2), 379-97
CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 119:49648

TI Contributions to the chemistry of boron. 214. Synthesis and reaction
chemistry of aminophosphinoboranes

IT Ring closure and formation
(of aminophosphinoboranes)

IT Crystal structure
Molecular structure
(of aminophosphinoboranes, diphosphadiboretanes and
triphosphatriborinanes)

IT 148446-90-6P 148446-91-7P 148446-92-8P
RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
(preparation and crystal and mol. structure of)

IT 112438-36-5P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
(Reactant or reagent)
(preparation and reaction of, with lithium disilylphosphide)

IT 112438-35-4P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
(Reactant or reagent)
(preparation and reactions of)

IT 147875-93-2P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
(Reactant or reagent)
(preparation and thermal intramol. cyclization of)

IT 112438-37-6P 122501-56-8P 147875-75-0P 147875-76-1P 147875-77-2P
147875-78-3P 147875-79-4P 147875-80-7P 147875-81-8P 147875-82-9P
147875-83-0P 147875-84-1P 147875-86-3P 147875-87-4P 147875-89-6P
147875-90-9P 147875-91-0P 147875-92-1P 147875-95-4P
148446-93-9P 148446-94-0P 148446-95-1P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

IT 147875-88-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation, crystal and mol. structure, and intramol. cyclization of)

IT 147875-94-3P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation, crystal and mol. structure, and reaction of, with methanol)

IT 122528-73-8P 147875-85-2P
 RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation, crystal and mol. structure, and reaction of, with
 pentacarbonylchromium-trimethylamine complex)

IT 59610-41-2 62263-24-5
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with aminodichloroboranes)

IT 15228-26-9 15228-32-7
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with aminophosphinoboranes)

IT 868-30-4 1113-31-1 1139-65-7 6591-26-0 44873-49-6 79855-29-1
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with lithium phosphides)

AB Aminochlorophosphinoboranes (R₂N)B(Cl)P(SiMe₃)₂ and (R₂N)B(Cl)PH₂ are
 obtained from elimination reactions between aminochloroboranes and
 LiP(SiMe₃)₂ and LiPH₂, resp. Selected reaction chemical of
 (i-Pr₂N)B(Cl)[P(SiMe₃)₂] with NH₃, Me₃SiN₃, Cr(CO)
)₅·NMe₃, and W(CO)₅·NMe₃ is described.
 The azide (i-Pr₂N)B(N₃)P(SiMe₃)₂ is stable at 25°; however,
 thermolysis at 80° provides a novel six-membered ring compound
 [(i-Pr₂N)BN(SiMe₃)P(SiMe₃)₂]. The reaction of (Ph₂N)B(Cl)P(SiMe₃)₂ with
 LiP(SiMe₃)₂ produces the only isolable bis(phosphino)borane, while
 combination of (R₂N)B(Cl)PH₂ with LiPH₂·DME yields new
 diphosphadiboretanes [(R₂N)BPH]₂ (R₂N = i-Pr₂N, Ph₂N, tmp =
 2,2,6,6-tetramethylpiperidino) and triphosphatriborinanes [(R₂N)BPH]₃ [R₂N
 = (Me₃Si)₂N, Me₂N, Et₂N]. Two salts, [(i-Pr₂N)BP(H)B(i-
 Pr₂N)PLi·DME]₂ and [tmpBP(H)B(tmp)PLi·DME]₂ (DME = ethylene
 glycol di-Me ether) are also isolated. The results of mol. structure
 detns. for [(i-Pr₂N)BN(SiMe₃)P(SiMe₃)₂], [tmpBPH]₂, [tmpBPH]₂·
 Cr(CO)₅, [(Me₃Si)₂N]BPH]₃, [(Me₃Si)₂N]BPH]₃·
 Cr(CO)₅, (Ph₂N)B[P(SiMe₃)₂]₂, and [(i-Pr₂N)BP(H)B(i-
 Pr₂N)PLi·DME]₂ by x-ray crystallog. are discussed.

L6 ANSWER 11 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN
 ACCESSION NUMBER: 1992:511748 CAPLUS <<LOGINID::20080918>>
 DOCUMENT NUMBER: 117:111748
 ORIGINAL REFERENCE NO.: 117:19511a,19514a
 TITLE: Tetraphosphafulvalene dianions isoelectronic to
 tetrathiafulvalene dications

AUTHOR(S): Maigrot, Nicole; Ricard, Louis; Charrier, Claude;
 Mathey, Francois

CORPORATE SOURCE: Lab. Chim. Phosphore Met. Transition, Ec. Polytech.,
 Palaiseau, F-91128, Fr.

SOURCE: Angewandte Chemie (1992), 104(8), 1082-4 (See also
 Angew. Chem., Int. Ed. Engl., 1992, 31(8), 1031-2)
 CODEN: ANCEAD; ISSN: 0044-8249

DOCUMENT TYPE: Journal
 LANGUAGE: German
 OTHER SOURCE(S): CASREACT 117:111748

TI Tetraphosphafulvalene dianions isoelectronic to tetrathiafulvalene
 dications

IT Crystal structure
 Molecular structure
 (of tetraphosphafulvalene dianion and its reaction product with sulfur)

IT 114862-89-4
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (metalation of, with lithium or potassium, dianions by)

IT 142397-66-8P 142397-69-1P
 RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
 (preparation and crystal structure of)

IT 142397-61-3P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
 (preparation and mol. structure of)

IT 142397-60-2P
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
 (Reactant or reagent)
 (preparation and reaction of, with sulfur or iron complex)

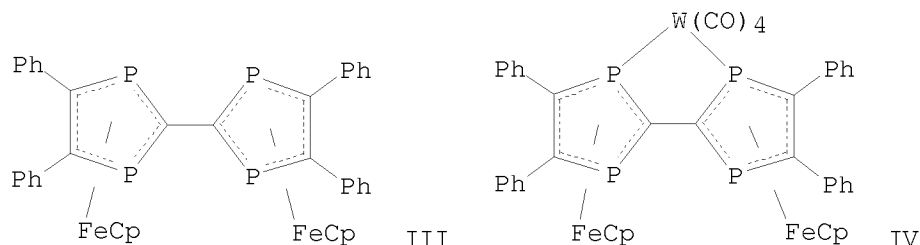
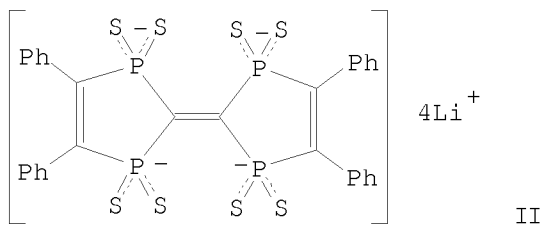
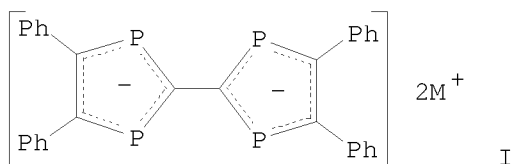
IT 142397-67-9P
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
 (Reactant or reagent)
 (preparation and reaction of, with tungsten complex)

IT 142397-59-9P 142397-68-0P
 RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation of)

IT 34978-37-5
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with tetraphosphafulvalene dianion)

IT 12129-25-8
 RL: RCT (Reactant); RACT (Reactant or reagent)
 (reaction of, with tetraphosphafulvalene dianion complex)

GI



AB Metalation of an octaphenyl derivative of tetraphosphafulvalene with Li or K metal in THF gave tetraphosphafulvalene dianions I (M = Li, K). Reaction of I (M = Li) with excess of sulfur gave lithium tetrakis(dithiophosphinate) derivative II. Reaction of I (M = Li) with (η⁵-cyclopentadienyl)(η⁶-xylene)iron hexafluorophosphate, [(Cp)(η⁶-1,4-Me₂C₆H₄) Fe]⁺ PF₆⁻, gave 30% iron complex III which on further reaction with [(nbd)W(CO)₄] (nbd = norbornadiene) gave tungsten complex IV. The crystal structures of I (M =

K) and II were determined I are isoelectronic to tetrathiafulvalene dications.

L6 ANSWER 12 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1991:66017 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 114:66017

ORIGINAL REFERENCE NO.: 114:11249a,11252a

TITLE: Structural hierarchy in M[6]T[4].vphi.n minerals

AUTHOR(S): Hawthorne, Frank C.

CORPORATE SOURCE: Dep. Geol. Sci., Univ. Manitoba, Winnipeg, MB, R3T 2N2, Can.

SOURCE: Zeitschrift fuer Kristallographie (1990), 192(1-2), 1-52

CODEN: ZEKRDZ; ISSN: 0044-2968

DOCUMENT TYPE: Journal

LANGUAGE: English

TI Structural hierarchy in M[6]T[4].vphi.n minerals

IT Minerals

RL: PRP (Properties)

(classification of, into crystal-structure hierarchy group based on bond valence theory)

IT 1302-58-5, Amblygonite 1318-31-6, Clinozoisite 1318-45-2, Dickite
1318-49-6, Epidote 1318-72-5, Kainite 1318-74-7, Kaolinite, properties
1318-81-6, Lawsonite 1319-12-6, Pumpellyite 1319-39-7, Roesslerite
1319-42-2, Zoisite 11089-72-8, Varulite-NaNa ((Mn1-1.5Fe0.5-1)MnNa(Na0.5-1Ca0-0.5)(PO4)3) 12004-28-3, Palermoite 12006-42-7, Arthurite
12025-97-7, Uklonskovite 12053-86-0, Linarite 12062-75-8, Tavorite
12068-50-7, Halloysite 12135-61-4, Titanite 12172-70-2, Allanite
12172-72-4, Alluaudite 12174-22-0, Piemontite 12198-53-7, Lazulite
12198-84-4, Natromontebasite 12206-95-0, Fibroferite 12211-29-9,
Ardenite 12211-42-6, Scorzalite 12218-55-2, Leucophosphite
12244-16-5, Endellite 12260-00-3, Childrenite 12260-04-7, Montebasite
12267-58-2, Paravauxite 12267-88-8, Isokite 12274-61-2,
Hagendorfite-NaNa ((Fe0.5-1Mg0-0.5)1-1.5Fe0.5-1]MnNa(Na0.5-1Ca0-0.5)(PO4)3) 12279-65-1, Nacrite 12284-84-3, Liroconite 12297-96-0,
Strunzite 12313-62-1, Gabrielsonite 12325-36-9, Mottramite
12337-04-1, Amarantite 12343-52-1, Metavauxite 12350-25-3, Mitridatite
12413-59-1, Apowite 12414-10-7, Bermanite 12415-36-0, Eosphorite
12416-31-8, Hancockite 12416-52-3, Holdenite 12417-43-5, Lacroixite
12417-80-0, Morinite 12417-91-3, Veszelyite 12418-01-8, Durangite
12418-22-3, Gordonite (Mg[Al(OH)(PO4)]2.8H2O) 12418-52-9,
Arsenosiderite 12418-83-6, Chlorothionite 12418-91-6, Butlerite
12418-92-7, Hohmannite 12419-94-2, Minyulite 12420-03-0, Moorhouseite
12420-18-7, Nickel hexahydrite 12421-00-0, Pseudolaueite 12424-68-9,
Sigloite 12426-43-6, Tilasite 12426-77-6, Parabutlerite 12518-72-8,
Stewartite 12601-23-9, Julgoldite 13587-33-2, Poitevinite
13759-09-6, Morenosite 13778-97-7, Hexahydrite 13817-21-5,
Chalcanthite 13824-49-2, Strengite 13824-50-5, Variscite 14457-55-7,
Epsomite 14567-64-7, Kieserite 14567-68-1, Szomolnokite 14567-70-5,
Siderotil 14567-75-0, Phosphosiderite 14567-77-2, Plancheite
14654-06-9, Clinoenstatite 14654-10-5, Phosphorrosslerite
(Mg(HPO4).7H2O) 14654-11-6, Newberyite 14654-14-9, Kolbeckite
14681-78-8, Orthoenstatite 14752-50-2, Metavariscite 15230-85-0,
Clinoferrosilite 15318-72-6, Laueite 15422-36-3, Orthoferrosilite
15490-91-2, Struvite 15491-15-3, Goslarite 15491-23-3, Melanterite
15553-21-6, Pentahydrite 15606-25-4, Dioptase 15652-21-8, Barbosalite
15669-02-0, Malayaite 16094-11-4, Conichalcite 16263-38-0, Austinite
17068-62-1, Hypersthene 19004-61-6, Descloizite 20775-42-2, Retgersite
20909-44-8, Scorodite 21793-55-5, Mansfieldite 22206-42-4,
Calciovolborthite 23540-24-1, Pyrobelonite 23625-76-5, Adelite
24189-48-8, Gunningite 24189-58-0, Clinohypersthene 24189-61-5,
Duftite 24250-37-1, Bieberite (Co(SO4).7H2O) 24250-39-3,

Bonattite 24250-46-2, Carminite 24250-50-8, Bianchite 24378-28-7,
 Boothite 24378-31-2, Starkeyite 33636-44-1, Tsumcorite 39473-77-3,
 Melonjosephite 51184-69-1, Jagowerite (Ba[Al(OH)(PO₄)]₂) 51198-90-4,
 Robertsite 51340-44-4, Khademite 52717-37-0, Szmikite 53125-64-7,
 Brassite 54652-49-2, Whitmoreite 58206-65-8, Foggite 59042-33-0,
 Krautite (Mn(HAsO₄).H₂O) 59492-33-0, Jurbanite 60867-12-1,
 Vuagnatite 61026-43-5, Bertossaite 64476-46-6, Rozenite 66183-78-6,
 Ferroxahydrite 66457-84-9, Ferropumpellyite 67712-23-6, Boyleite
 68108-22-5, Jokokuite (Mn(SO₄).5H₂O) 70315-78-5, Ilesite
 70878-67-0, Koritnigite 72276-48-3, Maghagendorfite-Na
 ([Mg_{0.5}-1Fe_{0.5}]-1.5Fe_{0.5}-1]Mn(Na_{0.5}-1Ca_{0.5})[(PO₄)₂(HPO₄)])
 73347-41-8, Mallardite 73729-55-2, Zinc-melanterite 74410-63-2,
 Fluckite 75137-72-3, Schmiederite 75797-73-8, Medaite 75797-77-2,
 Tiragalloite 79078-56-1, Cobalt-koritnigite 80147-36-0, Ojuelaite
 80487-65-6, Cechite 80487-70-3, Panasqueiraite 80487-72-5, Shuiskite
 83061-39-6, Arsendescloizite 83061-40-9, Kolfanite 83380-55-6,
 Dwornikite 85712-31-8, Ushkovite 88201-08-5, Ferrostrunzite
 93442-96-7, Earlshannonite 127030-85-7, Allanite-(Y)
 RL: PRP (Properties)

(classification of, into crystal-structure hierarchy group based on
 bond valence theory)

AB A large number of minerals have the general stoichiometry
 $A_x[M_6]T_4.vphi.n]y.vphi.z$, where A are large high-coordination number
 cations such as alkali metals and alkaline earths, M_{16} are [6]-coordinate
 divalent to quadrivalent cations, T_4 are [4]-coordinate trivalent to
 hexavalent cations, and .vphi. are unspecified simple anions. The square
 brackets denote the more strongly bonded part of the structure, called the
 structural unit. This way of expressing a structural formula essentially
 gives a binary representation of the structure, whereby the structural
 unit can be considered as a very complex oxyanion that interacts with the
 A species that constitute the cationic part of the structure; the .vphi.
 anions outside the square brackets are anions only very weakly held in the
 structure. There are 2 important features of expressing the formula in
 such a manner: very complex interactions within the structure are reduced
 to a simple binary interaction that is susceptible to quant. anal. using
 bond-valence theory; an hierarchical structural scheme may be set up by
 considering the graphical/topol. properties of the structural unit. Such
 a structure hierarchy is set up for minerals with structural units of the
 general formula $[M_6]T_4.vphi.n]$, based on the hypothesis that crystal
 structures may be ordered according to the polymerization of the coordination
 polyhedra of higher bond-valences. The structures are arranged into
 groups according to the dimension of polymerization of the structural unit and
 are arranged within these groups in terms of increasing d.p. There is
 definitely a preferred sequence of polyhedral linkage with increasing
 degree of condensation; from a completely disconnected structural unit,
 there is linkage between octahedra and tetrahedra, followed by linkage
 between octahedra, followed by linkage between tetrahedra. Along with
 this is a systematic change in the stoichiometry of the structural unit;
 this suggests that in a chemical formula, there is much more structural
 information than currently realized. The possible clusters of
 $[M_6]_2T_{41}2.vphi.n]$ stoichiometry are derived using graph theoretic and
 combinatorial techniques, subject to the boundary conditions that certain
 polyhedral linkages (e.g. face-sharing between tetrahedra) will not occur.
 There are 76 completely connected clusters of the form $[M_2T_2.vphi.n]$, but
 only 6 are found as fundamental building blocks in the structures of the
 96 minerals considered.

TITLE: Alkali metal phosphorus compounds and their reactions.
XXI. Reaction of potassium ethyl phosphide with dihaloalkanes

AUTHOR(S): Issleib, K.; Doell, G.

CORPORATE SOURCE: Martin-Luther Univ., HalleSaale, Germany

SOURCE: Zeitschrift fuer Anorganische und Allgemeine Chemie (1963), 324(5-6), 259-69
CODEN: ZAACAB; ISSN: 0044-2313

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

TI Alkali metal phosphorus compounds and their reactions. XXI. Reaction of potassium ethyl phosphide with dihaloalkanes

IT Insecticides
(dialkyl [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]phosphonates as)

IT Baptisia tinctoria
(glycosides of)

IT Paraffins
(α,ω -dihalo, reaction with (ethylphosphino)-potassium)

IT Ethyl ether, compound with [tetramethylenebis(ethylphosphinothioylidene)]dilithium
Phosphinodithioic acid, hexamethylenebis[ethyl-, cobalt salt
RL: PREP (Preparation)

IT 2404-55-9 93905-92-1
(Derived from data in the 7th Collective Formula Index (1962-1966))

IT 593-68-0, Phosphine, ethyl-
(potassium derivative, reaction with α,ω -dihaloalkanes)

IT 3040-70-8P, Tetraphosphetane, tetraethyl- 7789-43-7P, Cobalt bromide, CoBr₂, compds. with tetramethylenebis[ethylphosphine] 7789-43-7P, Cobalt bromide, CoBr₂, compds. with pentamethylenebis[ethylphosphine] 20626-76-0P, Tetraphosphetanium, 1,2,3,4-tetraethyl-1-methyl-, iodide 34616-20-1P, Phosphonic acid, [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]-, dimethyl ester 89798-26-5P, Phosphine, methylenebis[ethyl- 90274-10-5P, Phosphinodithioic acid, tetramethylenebis[ethyl- 90948-90-6P, Phosphonic acid, [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]-, diisopropyl ester 91065-37-1P, Phosphinodithioic acid, hexamethylenebis[ethyl- 91369-40-3P, Phosphonic acid, [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]-, dibutyl ester 91369-41-4P, Phosphonic acid, [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]-, diisobutyl ester 91469-51-1P, Phosphine sulfide, methylenebis[ethyl- 91772-46-2P, Phosphine, trimethylenebis[ethyl- 91772-47-3P, Phosphine sulfide, trimethylenebis[ethyl- 92045-94-8P, Phosphine, tetramethylenebis[ethyl- 92156-35-9P, Phosphonic acid, [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]-, diisopentylester 92381-19-6P, Phosphine, trimethylenebis[ethyl-, compound with HI 92381-19-6P, Hydriodic acid, compound with trimethylenebis[ethylphosphine] (2:1) 92400-37-8P, Phosphine, tetramethylenebis[ethyl-, compound with HI 92400-37-8P, Hydriodic acid, compound with tetramethylenebis[ethylphosphine] (2:1) 92443-15-7P, Phosphine sulfide, tetramethylenebis[ethyl- 92443-27-1P, Phosphine oxide, tetramethylenebis[ethyl- 93284-04-9P, Phosphine sulfide, pentamethylenebis[ethyl- 93284-52-7P, Phosphinodithioic acid, pentamethylenebis[ethyl-, nickel salt 93335-48-9P, Phosphine, pentamethylenebis[ethyl-, compound with HI 93456-93-0P, Phosphine, trimethylenebis[ethylmethyl-, compound with HI 93481-01-7P, Phosphine, pentamethylenebis[ethyl- 94073-52-6P, Hydriodic acid, compound with hexamethylenebis[ethylphosphine] 94073-52-6P, Phosphine, hexamethylenebis[ethyl-, compound with HI 94073-53-7P, Phosphine, tetramethylenebis[ethylmethyl-, compound with HI 94074-49-4P, Phosphine, hexamethylenebis[ethyl- 94115-74-9P, Phosphine, tetramethylenebis[ethyl-, compound with CoBr₂ 94315-15-8P, Phosphine, pentamethylenebis[ethyl-,

compound with CoBr₂ 95322-84-2P, Phosphine, hexamethylenebis[ethylmethyl-, compound with HI 96117-38-3P, Phosphine, pentamethylenebis[ethylmethyl-, compound with HI 97340-94-8P, Lithium, [trimethylenebis(ethylphosphinidene)]di-, compound with p-dioxane 98220-41-8P, Lithium, [tetramethylenebis(ethylphosphinidene)]di-, compound with p-dioxane 98338-80-8P, Lithium, [pentamethylenebis(ethylphosphinidene)]di-, compound with p-dioxane 105667-64-9P, Lithium, [hexamethylenebis(ethylphosphinidene)]di-, compound with p-dioxane 108652-58-0P, Lithium, [tetramethylenebis(ethylphosphinothioylidene)]di-, compound with Et₂O 820992-86-7P, Hydriodic acid, compound with pentamethylenebis[ethylphosphine] 859043-27-9P, Isopentyl alcohol, [2,2-dichloro-1-(dichloromethyl)-1-hydroxyethyl]phosphonate 859455-66-6P, Hydriodic acid, compound with tetramethylenebis[ethylmethylphosphine] (2:1) 860390-77-8P, Hydriodic acid, compound with trimethylenebis[ethylmethylphosphine] 860390-79-0P, Hydriodic acid, compound with pentamethylenebis[ethylmethylphosphine] 860390-81-4P, Hydriodic acid, compound with hexamethylenebis[ethylmethylphosphine]

RL: PREP (Preparation)
(preparation of)

IT 123-91-1P, p-Dioxane

RL: PREP (Preparation)

(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [hexamethylenebis(ethylphosphinidene)]dilithium (2:1))

IT 123-91-1P, p-Dioxane

RL: PREP (Preparation)

(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [pentamethylenebis(ethylphosphinidene)]dilithium (2:1))

IT 123-91-1P, p-Dioxane

RL: PREP (Preparation)

(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [tetramethylenebis(ethylphosphinidene)]dilithium (2:1))

IT 123-91-1P, p-Dioxane

RL: PREP (Preparation)

(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [trimethylenebis(ethylphosphinidene)]dilithium (2:1))

IT 88983-00-0, Potassium, (ethylphosphino)-

(reaction with α,ω -dihaloalkanes)

IT 7440-48-4, Cobalt

(salts, of hexamethylenebis[ethylphosphinodithioic acid])

IT 7440-02-0, Nickel

(salts, of pentamethylenebis[ethylphosphinodithioic acid])

IT 92443-15-7, Phosphine sulfide, tetramethylenebis[ethyl-

(P,P'-dilithium derivative, compound with Et₂O)

IT 91772-46-2, Phosphine, trimethylenebis[ethyl- 92045-94-8, Phosphine,

tetramethylenebis[ethyl- 93481-01-7, Phosphine, pentamethylenebis[ethyl- 94074-49-4, Phosphine, hexamethylenebis[ethyl-

(P,P'-dilithium derivative, compound with p-dioxane)

AB cf. CA 59, 5191f. Di-secondary phosphines, EtHP(CH₂)_nPHet (n = 3-6), chelate complex ligands, are formed by treatment of KPHEt with alkylene dihalides. These phosphines react with PhLi, MeI, and HI to form Et(Li)P[CH₂]_nP(Li)Et, [MeEtHP(CH₂)_nPHetMe]⁺⁺ 2I⁻, and [EtH₂P(CH₂)_nPH₂Et]⁺⁺ 2I⁻, resp. KPHEt (20.5 g.) was prepared from 20 g. EtPH₂ and 8.5 g. K in 500 ml. Pr₂O at 80-85°. A suspension of 20 g. KPHEt in Pr₂O was treated with 22 g. 1,6-dibromohexane in 500 ml. Pr₂O to yield 12 g. EtHP(CH₂)₆PHet, b₂ 92°. Similarly prepared were EtHP(CH₂)₅PHet (12.3 g.), b₂ 79° EtHP(CH₂)₄PHet (12.7 g.), b₁₆ 119°, and EtHP(CH₂)₃PHet (9.1 g.), b₁₇ 103°. Each of these di-secondary phosphines (2 g.) was dissolved in 20 ml. ether and treated with the corresponding amts. of MeI (3-4 g.) to give the resp. phosphonium salts, m. 110-12° 98-100°, 115-17° and 108-10°. The

same di-secondary phosphines (2 g.), each dissolved in 20 ml. ether, treated with 2 ml. concentrated aqueous HI gave corresponding phosphonium compds.,
 m. 99-110°, 86-8° 102-3° and 116-18°, resp.
 Solns. of the di-secondary phosphines (2 g.) in 5 ml. dioxane (C₄H₈O₂) were treated by dropwise addition of calculated amts. (16, 17, 19 and 20 ml., resp.) of PhLi solution (1 ml. = 94 mg.). The yields of colorless, difficultly soluble, extremely air- and moisture-sensitive products were: Et(Li)P(CH₂)₆P(Li)Et.₂C₄H₈O₂, 3.6 g.; Et(Li)P(CH₂)₅P(Li)Et.₂C₄H₈O₂, 3.3 g.; Et(Li)P(CH₂)₄P(Li)Et.₂C₄H₈O₂, 3.5 g.; and Et(Li)P(CH₂)₃P(Li)Et.₂C₄H₈O₂, 3.6 g. The following dithiophosphinic acids and phosphine sulfides were prepared by treating the corresponding di-secondary phosphines with various amts. of S in C₆H₆ solution: Et(HS)(S)P(CH₂)₆P(S)(SH)Et, m. 75-7°; EtH(S)P(CH₂)₆P(S)HET; EtH(S)P(CH₂)₄P(S)HET, m. 91-2°; Et(HS)(S)P(CH₂)₄P(S)(SH)Et, m. 125-7°; EtH(S)P(CH₂)₃P(S)HET, m. 86-7°. KPHEt (20 g.) in 500 ml. Pr₂O treated with 8 g. CH₂Cl₂ in 50 ml. Pr₂O gave only 0.7 g. EtHPCH₂PhEt₆, b₅ 54°. Other derivs. prepared for characterizing the various products were: [Et(S)(S)P(CH₂)₆P(S)(S)Et]--Ni⁺⁺, decomposed 200° Et(Li)(S)P(CH₂)₄P(S)(Li)Et.₄C₄H₁₀O; [Et(S)(S)P(CH₂)₆P(S)(S)Et]--Co⁺⁺; EtH(O)P(CH₂)₄P(O)HET, m. 100-2°; EtHP(CH₂)₄PHET.CoBr₂, decomposed 112-15°; EtHP(CH₂)₅PHET.CoBr₂, decomposed 118-20°; (PEt)₄, b₂ 164-6°; [(PEt)₄Me]I, m. 84-6°; EtH(S)PCH₂P(S)HET, m. 150-3°.

L6 ANSWER 14 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1963:428636 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 59:28636

ORIGINAL REFERENCE NO.: 59:5191f-h,5192a-d

TITLE: Alkali metal phosphorus compounds and their reactions. XX. 1,2-Ethylenebis(monocyclohexylphosphine) and -monoethylphosphine

AUTHOR(S): Issleib, Kurt; Doell, Gerhard

CORPORATE SOURCE: Martin-Luther-Univ., Halle, Germany

SOURCE: Chemische Berichte (1963), 96, 1544-50

CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 59:28636

TI Alkali metal phosphorus compounds and their reactions. XX.

1,2-Ethylenebis(monocyclohexylphosphine) and -monoethylphosphine

IT Alkali metal compounds

(phosphorus-containing organic)

IT Ethyl ether, compound of, with [ethylenebis(cyclohexylthiophosphinylidene)di lithium (4:1)]

Phosphine sulfide, ethylenebis[cyclohexyl-, ±-

RL: PREP (Preparation)

IT 18899-63-3, Phosphine, ethylenebis[cyclohexyl-

(P,P'-dilithium derivative)

IT 97001-21-3, Phosphine sulfide, ethylenebis[cyclohexyl-

(P,P'-dilithium derivative, compound with Et₂O)

IT 18899-62-2, Phosphine, ethylenebis[ethyl-

(P,P'-dilithium derivative, compound with p-dioxane)

IT 7723-14-0, Phosphorus

(compds., alkali metal derivs. of organic)

IT 1571-33-1P, Phosphonic acid, phenyl- 2359-99-1P, Diphosphine,

tetracyclohexyl- 3040-63-9P, Diphosphine, tetraethyl- 6411-21-8P,

Phosphine, ethylenebis[diethyl- 6710-67-4P, Diphosphonium, pentaethyl-,

iodide 7237-16-3P, Phosphonic acid, phenyl-, diisopropyl ester

18899-61-1P, Phosphine, ethylenebis[cyclohexylmethyl-, compound with HI

23743-26-2P, Phosphine, ethylenebis[dicyclohexyl- 29149-95-9P,

p-Diphosphorinane, 1,4-diethyl- 36540-03-1P, p-Diphosphorinane, 1,4-dicyclohexyl- 91773-78-3P, Phosphine sulfide, ethylenebis[ethyl- 92400-38-9P, Phosphine, ethylenebis[ethylmethyl-, compound with HI 94073-51-5P, Phosphine, ethylenebis[diethyl-, compound with HI 94626-87-6P, p-Diphosphorinane, 1,4-diethyl-, 1,4-disulfide 96419-23-7P, Lithium, [ethylenebis(cyclohexylphosphinidene)]di- 96471-96-4P, Lithium, [ethylenebis(ethylphosphinidene)]di-, compound with p-dioxane 97001-21-3P, Phosphine sulfide, ethylenebis[cyclohexyl-, meso- 98000-09-0P, p-Diphosphorinane, 1,4-dicyclohexyl-, 1,4-disulfide, cis- 98000-09-0P, p-Diphosphorinane, 1,4-dicyclohexyl-, 1,4-disulfide, trans- 98282-75-8P, p-Diphosphorinane, 1,4-dicyclohexyl-, 1,4-dioxide 100916-48-1P, Lithium, [ethylenebis(cyclohexylthiophosphinylidene)]di-, compound with Et₂O 860390-83-6P, Hydriodic acid, compound with ethylenebis[ethylmethylphosphine] 860390-85-8P, Hydriodic acid, compound with ethylenebis[diethylphosphine] 860390-87-0P, Hydriodic acid, compound with ethylenebis[cyclohexylmethylphosphine]

RL: PREP (Preparation)

(preparation of)

IT 123-91-1P, p-Dioxane

RL: PREP (Preparation)

(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [ethylenebis(ethylphosphinidene)]dilithium (2:1))

AB cf. CA 58, 13414g. K mononocyclohexylphosphide (I) and KPHEt (II) react with (CH₂Cl)₂ to yield 1,2-ethylenebis(monocyclohexylphosphine) (III) and (EtPHCH₂)₂ (IV), resp., which with MeI or S yield the corresponding phosphonium salts or disulfides, resp. Compds. containing asym. P atoms could be separated in 2 cases into the racemic and meso forms. The presence of the P-H bond in III and IV is demonstrated by their IR spectra and by metalation. Di-Li phosphides of the type (RLiPCH₂)₂ react with (CH₂Cl)₂ to yield the corresponding 1,4-dialkyl-1,4-diphosphacyclohexanes, which with S give cis, trans isomeric IVa. Li dicyclo-hexylphosphide (V) (25 g.) in 300 cc. refluxing Et₂O treated dropwise with stirring with 6.1 g. (CH₂Cl)₂ in 50 cc. Et₂O gave 1.2 l. C₂H₄; the mixture cooled and filtered yielded 19 g. tetracyclohexyldiphosphine (VI), m. 169°. V (11 g.) in 80 cc. THF treated with cooling slowly with 2.7 g. (CH₂Cl)₂ at 0° gave 100 cc. C₂H₄ and 2 g. VI; the filtrate concentrated and diluted with 5 cc. EtOH yielded 6 g. 1,2-ethylenebis(dicyclohexylphosphine), m. 96-7°. Et₂PLi (15.7 g.) from 13.8 g. PhLi and 16 g. Et₂PH in 150 cc. refluxing Et₂O treated dropwise with stirring with 8.1 g. (CH₂Cl)₂ gave 900 cc. C₂H₄; the mixture cooled, filtered, concentrated, and distilled

yielded 6 g. (Et₂P)₂ [characterized with EtI h in Et₂O as (Et₂PPEt₃)I, m. 100°] and 5 g. (Et₂PCH₂)₂ (VII) (characterized with 60% HI as VII.2HI, m. 181°). I (40 g.) in 400 cc. heptane treated with stirring at -20° with 12 g. (CH₂Cl)₂ in 50 cc. heptane, heated 2 h. at 70-5°, diluted with 100 cc. H₂O, worked up, and the crude product chromatographed on Alcoa yielded 26 g. III, air-sensitive, b₄ 162-8°. II (20 g.) in 500 cc. Pr₂O with 9 g. (CH₂Cl)₂ in 50 cc. Pr₂O gave 5 g. EtPH₂ and 6 g. IV, b₁₈ 90°. III (5.0 g.) and 6.0 g. MeI refluxed 5-10 min. in 50 cc. Et₂O, refrigerated several days, filtered, the residue dissolved in 250 cc. hot EtOH, and cooled gave 4.1 g. III.2 MeI, m. 300-1°; the filtrate concentrated to 10-15 cc. yielded 1.9 g. low-melting III.2MeI, m. 147-50°. IV (2.0 g.) in 20 cc. Et₂O and 4.0 g. MeI kept 4-5 h. at room temperature, the Et₂O decanted, and the residue recrystd. from 60 cc. EtOH gave 3.8 g. IV.2MeI, m. 158-60°. III (12.5 g.) in 100 cc. C₆H₆ and 3.1 g. S kept several hrs., shaken with 50 cc. 2N NaOH, and the aqueous phase acidified with dilute H₂SO₄ yielded 1.9

g.

1,2-ethylenebis(cyclohexyldithiophosphinic acid); the C₆H₆ phase concentrated, and the residue fractionally recrystd. from Me₂CO gave 4.9 g. dl1,2-ethylenebis(cyclohexylphosphine sulfide) (VIII), m. 155-8°,

and 1.9 g. the meso isomer, m. 125-30°. IV (2.0 g.) and 0.9 g. S in 20 cc. C₆H₆ yielded 1.8 g. [CH₂P(S)HEt]₂, m. 109-10° (aqueous Me₂CO). VIII (218.7 g.) treated with 5 cc. MeLi-Et₂O containing 156.5 mg. MeLi gave 30 cc. CH₄; a similar run with 212.8 mg. VIII yielded 29.5 cc. CH₄; the reaction mixture filtered gave the solid di-Li derivative of VIII with 4 mol Et₂O of crystallization III (6.0 g.) treated with cooling with 50 cc.

PhLi-Et₂O

(73 mg. PhLi/cc.) gave 5.6 g. di-Li derivative (IX) of III. IV (2.0 g.) in 5 cc. dioxane and 22 cc. PhLi-Et₂O (94 mg. PhLi/cc.) yielded 4 g. di-Li derivative (X) of IV. IX (8.1 g.) in 50 cc. refluxing Et₂O treated dropwise with 3 g. (CH₂Cl)₂ in 20 cc. Et₂O, refluxed 2 h., diluted with 10 cc. dioxane, filtered through kieselguhr, and distilled gave 1,4-dicyclohexyl-1,4-diphosphacyclohexane (XI), b₂ 225-30°. X (12.5 g.) in 30 cc. Et₂O and 4.0 g. (CH₂Cl)₂ in 20 cc. Et₂O gave 3.8 g. 1,4-di-Et analog (XII) of XI, light yellow, air-sensitive oil, b₄ 135-45°. XI (2.0 g.) and 0.45 g. S in 21 cc. C₆H₆ refluxed 10 min. and evaporated, and the residue crystallized from 15 cc. hot EtOH gave 0.8 g. IVa (R = cyclohexyl), m. 325 6°; the filtrate treated with 1 cc. H₂O gave 0.18 g. low-melting form, m. 250 5° (C₆H₆). XII (2.0 g.) and 0.73 g. S in 20 cc. C₆H₆ evaporated gave similarly 0.1 g. IVa (R = Et), m. 225 35°. XI (2.0 g.) and 20 cc. 3% H₂O₂ shaken 2-3 h. and evaporated, and the residue resuspended in EtOH and filtered off gave 1.7 g. 1,4-dioxide of XI, m. 260-75°. PHCH₂CH₂PH (11I) R (S) (S)R (IVa)

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ACCESSION NUMBER: 1962:31535 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 56:31535

ORIGINAL REFERENCE NO.: 56:5996g-i,5997a-e

TITLE: Alkali metal-phosphorus compounds and their reactions. XI. The reaction of potassium cyclohexylphosphide with dihaloalkanes

AUTHOR(S): Issleib, Kurt; Doell, Gerhard

CORPORATE SOURCE: Univ. Halle, Germany

SOURCE: Chemische Berichte (1961), 94, 2664-9
CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

TI Alkali metal-phosphorus compounds and their reactions. XI. The reaction of potassium cyclohexylphosphide with dihaloalkanes

IT Reactivity
(of phosphorus compds. with alkali metals)

IT Alkylene halides
(reaction with (cyclohexylphosphino)lithium)

IT Lithium, [hexamethylenebis(cyclohexylphosphinidene)]di-, compound with p-dioxane
Lithium, [tetramethylenebis(cyclohexylphosphinidene)]di-
RL: PREP (Preparation)

IT 65105-85-3 107712-89-0 108652-55-7
(Derived from data in the 7th Collective Formula Index (1962-1966))

IT 75-15-0, Carbon disulfide
(compds. with tetramethylenebis[cyclohexylmethylphosphine])

IT 7723-14-0, Phosphorus
(compds., alkali metal derivs. of organic)

IT 822-68-4P, Phosphine, cyclohexyl- 3040-71-9P, Tetraphosphetane, tetracyclohexyl- 3040-77-5P, Phosphine, cyclohexyl-, compound with HI 88591-62-2P, Phosphinodithioic acid, tetramethylenebis[cyclohexyl- 90050-09-2P, Phosphine, cyclohexylmethyl- 90114-83-3P, Phosphine, tetramethylenebis[cyclohexyl- 90229-07-5P, Phosphinodithioic acid, tetramethylenebis[cyclohexyl-, nickel salt 91725-33-6P, Phosphine, cyclohexyl-, compound with HBr 92799-67-2P, Phosphine, cyclohexyldimethyl-, compound with HI 94376-93-9P, Phosphinodithioic acid,

hexamethylenebis[cyclohexyl- 97340-94-8P, Lithium,
 [trimethylenebis(ethylphosphinidene)]di-, compound with p-dioxane
 97472-13-4P, Phosphine, trimethylenebis[cyclohexyl- 98176-10-4P,
 Lithium, [pentamethylenebis(cyclohexylphosphinidene)]di- 98283-36-4P,
 Phosphine, trimethylenebis[cyclohexylmethyl-, compound with HI
 98341-94-7P, Phosphine, pentamethylenebis[cyclohexyl- 98884-44-7P,
 Phosphine, tetramethylenebis[cyclohexylmethyl-, compound with HI
 99080-60-1P, Phosphine, pentamethylenebis[cyclohexylmethyl-, compound with
 HI 99729-90-5P, Phosphine, hexamethylenebis[cyclohexylmethyl-, compound
 with HI 99812-62-1P, Phosphine, tetramethylenebis[cyclohexylmethyl-,
 compound with CS₂ 99926-44-0P, Phosphine, hexamethylenebis[cyclohexyl-
 101016-80-2P, Phosphinic acid, hexamethylenebis[cyclohexyl-
 108271-78-9P, Lithium, [trimethylenebis(cyclohexylphosphinidene)]d
 i-, compound with p-dioxane
 RL: PREP (Preparation)

(preparation of)

IT 123-91-1P, p-Dioxane

RL: PREP (Preparation)

(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of,
 with [hexamethylenebis(cyclohexylphosphinidene)]dilithium)

IT 123-91-1P, p-Dioxane

RL: PREP (Preparation)

(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of,
 with [trimethylenebis(cyclohexylphosphinidene)]dilithium)

IT 123-91-1P, p-Dioxane

RL: PREP (Preparation)

(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of,
 with [trimethylenebis(phenylphosphinidene)]dilithium)

IT 93386-85-7, Potassium, (cyclohexylphosphino)-

(reaction with alkylene halides)

IT 28240-66-6, Phosphine, trimethylenebis[phenyl-

(P,P'-dilithium derivative, compound with p-dioxane)

AB K cyclohexylphosphide (I) with 1,3-, 1,4-, 1,5-, and 1,6-dihaloalkanes
 yields the corresponding C₈H₁₁PH(CH₂)_nPHC₆H₁₁ (n = 3-6) (II). I with
 (CH₂Br)₂ or CH₂Cl₂ yields in addition to C₂H₄, cyclohexylphosphine (III), and
 tetracyclohexylcyclotetraphosphine (IV), because of a metal-halogen
 exchange, also methylcyclohexylphosphine (V) and IV. The 2 H
 atoms of the II are replaceable by Li to yield the corresponding di-Li
 derivs. (VI). The preparation of phosphonium salts and dithio acids of the
 types [Me(C₆H₁₁)HP(CH₂)_nPH(C₆H₁₁)Me]I₂ (VIa) and
 C₆H₁₁(HS)(S)P(CH₂)_nP(S)(SH)C₆H₁₁ (n = 3-6) (VII), resp. The reaction of
 III with (CH₂Br)₂ yields by halogen-H exchange IV, III.HBr, and C₂H₄. III
 (35 g.), 500 cc. heptane, and 7.5 g. K refluxed 4-6 hrs. with stirring,
 cooled, and filtered gave 39 g. I, air- and moisture-sensitive solid. I
 in 300 cc. refluxing C₆H₆ treated during 1 hr. with an appropriate
 dihaloalkane in 100 cc. C₆H₆, refluxed 0.5 hr., and decomposed with 50 cc.
 O-free H₂O, and the C₆H₆ layer worked up gave the corresponding II. I (20
 g.) and 9.5 g. Cl(CH₂)₆Cl gave 13.5 g. II (n = 6) (VIII), b₂
 200-1°, m. 13-14°. I (20 g.) and 14 g. Br(CH₂)₅Br yielded
 11.8 g. II (n = 5) (IX), b₂ 177°. I (20 g.) and 8 g. Cl(CH₂)₄Cl
 gave 11.7 g. II (n = 4) (X), b₂ 165-6°, m. 4°. I (20 g.)
 and 12.5 g. Br(CH₂)₃Br yielded 10.5 g. II (n = 3) (XI), b₂ 151°.
 The appropriate II in 25 cc. Et₂O treated with excess MeI, kept several
 hrs. and filtered gave the corresponding VIa (n, m.p., g. yield, g. amount
 and II used are given): 6, 158-60°, 2.9, 2, VIII; 5, 160-2°,
 2.3, 2, IX; 4 (XII), 185-7°, 2.7, 2, X; 3, 133-5°, 2.3, 2,
 XI. The appropriate II in 25-30 cc. Et₂O treated dropwise with the calculated
 amount of PhLi in Et₂O and filtered without or with previous addition of
 dioxane gave the following di-Li derivs. (n, number of moles of complexed
 dioxane, g., amount II, and cc. volume dioxane used and g., yield of product
 are given): 6, 2, 2, 3, 3.1; 5, 0, 2, 0, 2; 4, 0, 2, 0, 2; 3, 2, 2, 3,

3.1. VIII (2 g.) in 25 cc. C₆H₆ treated with 0.8 g. S, boiled briefly, and evaporated, and the oily residue dissolved in aqueous NaOH, filtered, and acidified gave 1.6 g. VII (n = 6), m. 108-10° (aqueous MeOH). X (5 g.) and 2.2 g. S gave similarly 6.1 g. VII (n = 4) (XIII), m. 156-7°. VIII (2 g.) in 50 cc. C₆H₆ treated with a stream of air until evaporated gave 1.9 g. 1,6-hexamethylenebis(cyclohexylphosphinic acid), m. 140-2° (PhMe). XII (1 g.), 10 cc. H₂O, 20 cc. Et₂O, and 0.5 g. NaOH shaken briefly, and the Et₂O layer dried and treated with 1-2 cc. CS₂ gave 0.59 g. pale red 1,4-butylenebis(methylcyclohexylphosphine)-CS₂, adduct, which decomposed gradually. XIII (1.5 g.), 0.5 g. NiBr₂, and 25 cc. C₆H₆ refluxed 5 hrs. and filtered yielded 1 g. Ni salt of XIII, decomposed 355°. III (10 g.) and 8 g. (CH₂Br)₂ refluxed 2 hrs., triturated with hot EtOH, and recrystd. from C₆H₆ gave 1.6 g. IV, m. 220°; 280 cc. C₂H₄ was evolved during the reaction; III.HBr, m. 143-5°, had sublimed during the reaction into the condenser; it decomposed in EtOH or in air into III and HBr. I (10 g.) in 250 cc. hot C₆H₆ treated dropwise with stirring with 6 g. (CH₂Br)₂ in 50 cc. C₅H₆ gave 610 cc. C₂H₄; the mixture treated with 20 cc. O-free H₂O, the C₆H₆ layer evaporated, and the residue diluted with 100 cc. Et₂O gave 2.9 g. IV, leaflets, m. 220°; the Et₂O filtrate concentrated to 10-20 cc. and treated with HI gave III.HI, leaflets,

m.

170° (EtOH). I (10 g.) and 2.7 g. CH₂Cl₂ in 250 cc. C₆H₆ gave similarly 1.7 g. IV, m. 220°; the Et₂O layer gave 2 g. V, b₃ 30-1°. V (1 g.) and 2 g. MeI in 15 cc. Et₂O yielded 1 g. dimethylcyclohexylphosphonium iodide, m. 140-2° (EtOH). I (10 g.) in 250 cc. C₆H₆ treated dropwise with stirring with 5.2 g. Br in 50 cc. C₆H₆, filtered, concentrated, and diluted with Et₂O yielded 2.6 g. IV, m. 220°; the filtrate concentrated and treated with HI gave III.HI, m. 170°.

L6 ANSWER 16 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1962:31534 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 56:31534

ORIGINAL REFERENCE NO.: 56:5995f-i, 5996a-g

TITLE: Alkali metal-phosphorus compounds and their reactions. X. Alkali phosphides of the type Ar(Li)P(CH₂)_nP(Li)Ar and their reaction with alkyl and cycloalkyl halides

AUTHOR(S): Issleib, Kurt; Krech, Frieder

CORPORATE SOURCE: Univ. Halle, Germany

SOURCE: Chemische Berichte (1961), 94, 2656-63

CODEN: CHBEAM; ISSN: 0009-2940

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 56:31534

TI Alkali metal-phosphorus compounds and their reactions. X. Alkali phosphides of the type Ar(Li)P(CH₂)_nP(Li)Ar and their reaction with alkyl and cycloalkyl halides

IT Reactivity

Reactivity

(of phosphorus compds. with alkali metals)

IT Alkylene halides

(reaction with (cyclohexylphosphino)lithium)

IT Alkyl halides

(reaction with phosphine derivs.)

IT Lithium, [pentamethylenebis(phenylphosphinidene)]di-, compound with p-dioxane

RL: PREP (Preparation)

IT 100321-06-0 107712-89-0 108486-25-5 108652-55-7

(Derived from data in the 7th Collective Formula Index (1962-1966))

IT 7723-14-0, Phosphorus

(compds., alkali metal derivs. of organic)

- IT 3302-87-2P, Phospholane, 1-phenyl- 6050-22-2P, 1,2-Diphospholane, 1,2-diphenyl- 25900-28-1P, Phosphine sulfide, trimethylenebis[ethylphenyl- 25900-30-5P, Phosphine sulfide, tetramethylenebis[cyclohexylphenyl- 25900-32-7P, Phosphine sulfide, pentamethylenebis[ethylphenyl- 28240-67-7P, Phosphine, tetramethylenebis[phenyl- 65105-85-3P, 1,2-Diphospholane, 1,2-diphenyl-, 1,2-disulfide 72144-83-3P, Phosphine, tetramethylenebis[cyclohexylphenyl- 73191-51-2P, Phosphine, hexamethylenebis[phenyl- 83152-25-4P, Phosphine, hexamethylenebis[ethylphenyl- 89399-68-8P, Phosphine sulfide, pentamethylenebis[cyclohexylphenyl- 90116-42-0P, Phosphine, pentamethylenebis[cyclohexylphenyl- 97340-94-8P, Lithium, [trimethylenebis(ethylphosphinidene)]di-, compound with p-dioxane 98170-74-2P, Phosphine, hexamethylenebis[cyclohexylphenyl- 98341-81-2P, Phosphine, pentamethylenebis[phenyl- 99888-09-2P, Phosphine, trimethylenebis[ethylphenyl- 100151-95-9P, Phosphine, tetramethylenebis[ethylphenyl- 100151-96-0P, Phosphine sulfide, tetramethylenebis[ethylphenyl- 100170-44-3P, Phosphine oxide, tetramethylenebis[ethylphenyl- 100335-42-0P, Nickel, dibromo[pentamethylenebis[ethylphenylphosphine]]- 100407-14-5P, Phosphine, pentamethylenebis[ethylphenyl- 100433-15-6P, Phosphine, pentamethylenebis[(p-ethylphenyl)-, nickel complex 100776-08-7P, Lithium, [hexamethylenebis(phenylphosphinidene)]di-, compound with p-dioxane 100997-04-4P, Phosphine sulfide, hexamethylenebis[ethylphenyl-, stereoisomers 101320-02-9P, Phosphonium, tetramethylenebis[diethylphenyl- iodide] 106524-86-1P, Phosphine, pentamethylenebis[phenyl-, compound with HI 106572-33-2P, Phosphine sulfide, hexamethylenebis[cyclohexylphenyl- 107101-27-9P, Phosphine oxide, tetramethylenebis[cyclohexylphenyl- 108271-53-0P, Lithium, [tetramethylenebis(phenylphosphinidene)]di-, compound with p-dioxane
RL: PREP (Preparation)
(preparation of)
- IT 123-91-1P, p-Dioxane
RL: PREP (Preparation)
(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [hexamethylenebis(phenylphosphinidene)]dilithium)
- IT 123-91-1P, p-Dioxane
RL: PREP (Preparation)
(reaction with (diphenylstibino)lithium (1:1), preparation and reactions of, with [tetramethylenebis(phenylphosphinidene)]dilithium)
- IT 28240-66-6, Phosphine, trimethylenebis[phenyl- 28240-67-7, Phosphine, tetramethylenebis[phenyl- 73191-51-2, Phosphine, hexamethylenebis[phenyl- (P,P'-dilithium derivative, compound with p-dioxane)
- IT 98341-81-2, Phosphine, pentamethylenebis[phenyl- (P,P'-dilithium derivative, compds. with p-di-oxane)
- IT 822-68-4, Phosphine, cyclohexyl- (P-potassium derivative, reaction with alkylene halides)
- AB cf. CA 55, 15428a.-Dissecondary phosphines of the general formula $\text{ArHP}(\text{CH}_2)_n\text{PAr}$ ($n = 3-6$) with PhLi yield the corresponding di-Li phosphides which with alkyl and cycloalkyl halides give unsym. substituted phosphines. $\text{Ph}(\text{Li})\text{P}(\text{CH}_2)_3\text{P}(\text{Li})\text{Ph}$ (I) yields with EtI , in addition to $[\text{EtPhPCH}_2]_2\text{CH}_2$ (II), because of a metal-halogen exchange 1,2-diphenyl-1,2-diphosphacyclopentane (III) which can also be obtained from I and $(\text{CH}_2\text{Br})_2$. The unsym. ditertiary phosphines can be converted to the corresponding disulfides, bisphosphonium salts, or dioxides by reaction with S, alkyl halides, or by oxidation, resp. NaPPh (IV) (50 g.) in 200 cc. Et_2O treated dropwise with stirring with 43.5 g. $\text{Br}(\text{CH}_2)_5\text{Br}$, refluxed 0.5 hr., cooled, diluted with 150 cc. H_2O , and worked up gave 43.5 g. air-sensitive $[\text{PhPH}(\text{CH}_2)_2]_2\text{CH}_2$ (V), b₄₋₅ 211-14°. V (6 g.) in 150 cc. Et_2O saturated with dry HI and filtered gave 4.1 g. V.HI, m. 155-7° (repptd. from EtOH with Et_2O). IV (48.5 g.) treated with 28.5 g. $\text{Cl}(\text{CH}_2)_6\text{Cl}$ in 170 cc. Et_2O yielded 48 g.

[PhHP(CH₂)₃]₂ (VI), b₄ 207-11°. IV (56.2 g.) in 200 cc. Et₂O and 27 g. Cl(CH₂)₄Cl gave similarly 12.5 g. 1-phenylcyclotetramethylenephosphine, b₄ 110-12°, 29.6 g. [PhHP(CH₂)₂]₂, b₄ 195-7°, and 5.6 g. PhPH₂. VI (1.65 g.) in 15 cc. Et₂O treated dropwise with 15 cc. PhLi solution containing 61 mg./cc., kept several hrs., added with stirring to 10

cc.

dioxane, and filtered yielded 2.3 g. [Ph(Li)P(CH₂)₃]₂.2C₄H₈O₂ (VII). V (13.4 g.) in 150 cc. Et₂O with 85 cc. PhLi-Et₂O containing 92 mg./cc. and 30 cc. dioxane refluxed 0.5 hr. yielded similarly 27.3 g. [Ph(Li)P(CH₂)₂]₂.2C₄H₈O₂ (VIII). V (0.8 g.) in 20 cc. Et₂O, 50 cc. PhLi-Et₂O (93.3 mg./cc.), and 5 cc. dioxane gave 1.2 g. [Ph(Li)P(CH₂)₂]₂.2C₄H₈O₂. VIII heated 5 hrs. at 100° gave [Ph(Li)P(CH₂)₂]₂.2C₄H₈O₂. [PhHP(CH₂)₂]₂ (2.1 g.) in 120 cc. Et₂O with 14 cc. PhLi-Et₂O (92 mg./cc.) and 10 cc. dioxane gave 2.8 g. [Ph(Li)P(CH₂)₂]₂.2C₄H₈O₂ (VIIIa). VII (12.5 g.) in 200 cc. Et₂O treated dropwise with 12.4 g. EtI in 50 cc. Et₂O, refluxed 0.5 hr., and diluted with 100 cc. H₂O, and the Et₂O layer worked up gave 8.3 g. [EtPhP(CH₂)₃]₂ (IX), needles, m. 40-1°, b_{0.1} 193-6°. IX (2.84 g.) in 20 cc. C₆H₆ refluxed briefly with 0.51 g. S, the C₆H₆ distilled, and the residue treated 24 times with 10-cc. portions MeOH left 1.04 g. (crude) meso-disulfide of IX, needles, m. 163-4° (Me₂CO); the MeOH extract evaporated gave 1.65 g. racemic disulfide of IX, m. 88-90°. VII (26 g.) in 200 cc. PhMe treated with stirring with 17.3 g. cyclohexyl bromide in 20 cc. PhMe, boiled briefly, filtered, and evaporated gave 20.6 g. 1,6-hexamethylenebis(cyclohexylphenylphosphine) (X), viscous oil. X (2 g.) in 50 cc. C₆H₆ treated with 0.3 g. S gave 0.3 g. disulfide of X, m. 211-14° (C₆H₆). VIII (16.7 g.) and 17.3 g. EtI in 225 cc. Et₂O treated with 10 cc. dioxane, filtered, and distilled yielded 12 g. [EtPhP(CH₂)₂]₂ (XI), b₃ 211-13°. XI (2 g.), 0.4 g. S, and 20 cc. C₆H₆ gave 0.5 g. disulfide of XI, m. 115-16° (MeOH). XI (2 g.), 3 g. NiBr₂, and 35 cc. PhMe refluxed 2 hrs. yielded 2.1 g. dibromo-1,5-pentamethylenebis(ethylphenylphosphine)nickel, red-brown, m. 148-53° (PhMe). VIII (25.5 g.), 13.5 g. cyclohexyl bromide, and 230 cc. PhMe gave similarly an oily product which diluted with 150 cc. hot EtOH, filtered, and cooled yielded 14.8 g. 1,5-pentamethylenebis(cyclohexylphenylphosphine) (XII), oil at room temperature

XII

(3.02 g.), 0.43 g. S, and 35 cc. C₆H₆ yielded in the usual manner 0.43 g. disulfide of XII, needles, m. 184-5° (Me₂CO). VIIIa (30.9 g.), 33.7 g. EtI, and 300 cc. Et₂O gave in the usual manner 22.4 g. (EtPhPCH₂CH₂)₂ (XIII), b₄ 225-30°. XIII (2.7 g.), 0.6 g. S, and 50 cc. C₆H₆ gave 0.5 g. disulfide of XIII, needles, m. 179-80° (Me₂CO). XIII (2 g.) in 20 cc. Me₂CO treated with KMnO₄-Me₂CO until the color persisted, decolorized with a few drops 3% aqueous H₂O₂, filtered, and evaporated gave 0.25 g. [EtPhP(O)CH₂CH₂]₂, m. 159-61° (PhMe). XIII (2 g.) and 3 g. EtI in 40 cc. EtOH refluxed 0.5 hr. and refrigerated gave 2.9 g. [(Et₂PhPCH₂CH₂)₂]₂, m. 210-12° (EtOH). VIIIa (12.3 g.) and 6.3 g. cyclohexyl chloride in 170 cc. dioxane gave similarly 7 g. 1,4-tetramethylenebis(cyclohexylphenylphosphine) (XIV), m. 104-5°. XIV (1.5 g.), 0.3 g. S, and 50 cc. C₆H₆ yielded 0.36 g. disulfide of XIV, m. 248-50°. XIV (4.9 g.) oxidized in the usual manner with KMnO₄ gave 0.76 g. 1,4-tetramethylenebis(cyclohexylphenylphosphine oxide), m. 201-2°. I (11.5 g.) in 200 cc. Et₂O treated dropwise with EtCl in Et₂O, shaken immediately with two 100-cc. portions H₂O, and worked up gave 9.2 g. II, b₃ 196-200°. II (2.13 g.), 0.43 g. S, and 30 cc. C₆H₆ gave 0.56 g. disulfide of II, m. 139-40°. I.2C₄H₈O₂ (24.6 g.) in 200 cc. C₆H₆ and 20 cc. tetrahydrofuran treated during 3 hrs. with 10.3 g. (CH₂Br)₂ in 100 cc. C₆H₆ gave about 1110 cc. C₂H₄; the mixture filtered and evaporated gave 8.8 g. 1,2-diphenyl-1,2-diphosphacyclopentane (XV), b₄ 184-90°. XV (2.83 g.) and 0.7 g. S in 15 cc. C₆H₆ refluxed a few min. and evaporated yielded 1.74 g. 1,2-diphenyl-1,2-diphosphacyclopentane

1,2-disulfide, m. 178-80° (Me2CO).

L6 ANSWER 17 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1962:31533 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 56:31533

ORIGINAL REFERENCE NO.: 56:5995e-f

TITLE: Reaction of phosphine and of phosphorous acid with aldehydes. V. Reaction of hypophosphorous acid with formaldehyde and benzaldehyde

AUTHOR(S): Horak, J.; Ettel, V.

CORPORATE SOURCE: Inst. Chem. Technol., Prague

SOURCE: Collection of Czechoslovak Chemical Communications (1961), 26, 2410-17

CODEN: CCCCAK; ISSN: 0010-0765

DOCUMENT TYPE: Journal

LANGUAGE: German

TI Reaction of phosphine and of phosphorous acid with aldehydes. V.

Reaction of hypophosphorous acid with formaldehyde and benzaldehyde

IT Aldehydes

(reactions of, with P compds.)

IT 100321-06-0 108486-25-5

(Derived from data in the 7th Collective Formula Index (1962-1966))

IT 90-09-5P, Phosphinic acid, bis(α -hydroxybenzyl)- 2074-67-1P,
Phosphinic acid, bis(hydroxymethyl)- 52705-43-8P, Phosphonous acid,
(α -hydroxybenzyl)- 60672-77-7P, Phosphonous acid, (hydroxymethyl)-

RL: PREP (Preparation)

(preparation of)

IT 7803-51-2, Phosphine 13598-36-2, Phosphorous acid

(reaction with aldehydes)

IT 7803-51-2, Phosphine

(reactions of derivs. of, with aldehydes)

IT 50-00-0, Formaldehyde

(reactions of, with hypophosphorous acid)

IT 100-52-7, Benzaldehyde

(reactions of, with hypophosphorous acid, kinetics of)

IT 6303-21-5, Hypophosphorous acid

(reactions with HCHO or BzH, kinetics of)

AB The products of the reactions mentioned in the title have the structures: RCHOHPO2H2 (I), RCHOHPO(OH)CHOHR (II), where R stands for H or Ph. The reaction rate is linearly proportional to the aldehyde and undissocd. H3PO2 concns. For the sake of simplicity it is possible to express the rate equations in the following way: $v = k [\text{CH}_2\text{O}]^{0.7} [\text{H}_3\text{PO}_2] (1 - \alpha)$ and $v = k [\text{PhCHO}]^{0.85} [\text{H}_3\text{PO}_2] (1 - \alpha)$, where α means the degree of the dissociation of H3PO2. The values of the velocity consts. at 90° are $0.295 \pm 0.023 \text{ l.0.7 mole}^{-0.7} \text{ hr.}^{-1}$ (CH2O) and $0.136 \pm 0.011 \text{ l.0.85 mole}^{-0.85} \text{ hr.}^{-1}$ (PhCHO). A reaction scheme is suggested.

L6 ANSWER 18 OF 18 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1959:121944 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 53:121944

ORIGINAL REFERENCE NO.: 53:21843i,21844a-i

TITLE: Synthesis of 2,3:6,7-dibenzodiphenylene, and reaction of 1-bromo-2-iodonaphthalene with magnesium

AUTHOR(S): Ward, E. R.; Pearson, B. D.

CORPORATE SOURCE: Leicester Coll. Technol. Commerce, UK

SOURCE: Journal of the Chemical Society (1959) 1676-80

CODEN: JCSOA9; ISSN: 0368-1769

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 53:121944

TI Synthesis of 2,3:6,7-dibenzodiphenylene, and reaction of
 1-bromo-2-iodonaphthalene with magnesium
 IT 811-20-1 108632-57-1 126329-92-8 128633-89-6
 129068-11-7
 (Derived from data in the 6th Collective Formula Index (1957-1961))
 IT 242-50-2P, 6H-Dibenzo[b,h]carbazole 252-44-8P, Dibenzo[a,g]biphenylene
 258-47-9P, Dibenzo[b,h]biphenylene 13115-28-1P, 2-Naphthylamine,
 3-nitro- 29492-74-8P, Acetamide, N-(5,6,7,8-tetrahydro-3-nitro-2-
 naphthyl)- 50537-06-9P, Naphthalene, 1,2,3,4-tetrahydro-6-iodo-7-nitro-
 102153-44-6P, Naphthalene, 2-bromo-3-iodo- 102153-71-9P, Naphthalene,
 2-iodo-3-nitro- 102317-09-9P, 2,2'-Binaphthyl, 5,5',6,6',7,7',8,8'-
 octahydro-3,3'-dinitro- 110939-85-0P, Dibenzo[b,h]biphenylene,
 tetrahydro- 112376-54-2P, 1,2,3,4,8,9,10,11-Octahydrodinaphth[2,3-
 b:2',3'-d]iodolium iodide 114164-69-1P, 3,3'-Bi[2-naphthylamine],
 5,5',6,6',7,7',8,8'-octahydro-
 RL: PREP (Preparation)
 (preparation of)
 IT 7439-95-4, Magnesium
 (reaction with 1-bromo-2-iodonaphthalene)
 IT 90948-03-1, Naphthalene, 1-bromo-2-iodo-
 (reaction with Mg)
 AB 2,3:6,7-Dibenzodiphenylene (I) was synthesized by the pyrolysis of
 5,6,7,8,5',6',7',8'-octahydro-2,2'-dinaphthyl-3,3'-iodonium iodide (II)
 and was accompanied by 2 other products which appear to be hydrogenated
 derivs. of I. The pyrolysis of the corresponding 3,3'-tetrazonium sulfate
 (III) was also studied. 1-Bromo-2-iodonaphthalene (IV) was prepared as
 follows. 1-Bromo-2-naphthylamine (10 g.) in 7 cc. warm H₂SO₄ and 20 cc.
 H₂O diazotized at 0° by rapid addition of 5 g. NaNO₂ in 15 cc. H₂O,
 the solution added to 6 g. iodine and 18 g. KI in 120 cc. H₂O, stirred 1 hr.,
 the solids collected, shaken with 200 cc. 10% aqueous Na₂S₂O₃, collected
 again, washed, dried, dissolved in C₆H₆, and passed through Al₂O₃ gave 6
 g. IV, m. 94°. 3-Bromo-2-naphthylamine (10 g.) in 4 g. NaNO₂ in 40
 cc. H₂SO₄ stirred 1 hr. below 30° into 80 cc. AcOH, and the solution
 treated with iodine-KI gave 10 g. 2-bromo-3-iodonaphthalene, m.
 120° (alc.-EtOAc). 3-Nitro-2-naphthylamine (10 g.) in 120 cc. AcOH
 similarly gave 14 g. 2-iodo-3-nitronaphthalene, m. 89° (alc.).
 6-Acetamido-1,2,3,4-tetrahydronaphthalene (50 g.) in 810 cc. Ac₂O treated
 dropwise below 25° with 16.7 cc. Ac₂O and 16.7 cc. HNO₃, left
 overnight, poured into ice-H₂O, and the liquid siphoned off gave 35-9%
 6-acetamido-1,2,3,4-tetrahydro-7-nitronaphthalene (V). Further
 amts. were obtained by evaporation of the mother liquor to dryness and
 separation
 from the 5-nitro isomer by chromatography on Al₂O₃ in C₆H₆-EtOAc.
 Diazotizing V and adding the diazonium solution to aqueous iodine-KI
 underlaid with CHCl₃ gave 60-80% 1,2,3,4-tetrahydro-6-iodo-7-
 nitronaphthalene (VI). VI (1 g.) heated during 0.5 hr. to 132°
 with portionwise addition of 0.6 g. Cu-bronze, the temperature kept below
 140°, the heating continued another 0.5 hr., extracted with hot C₆H₆,
 and chromatographed on Al₂O₃ gave 0.42 g. 3,3'-dinitro-5,5',6,6',7,7',8,8'-
 octahydro-2,2'-binaphthyl (VII), m. 190-1°. VII (3.5 g.) in 165
 cc. EtOAc and 15 cc. alc. in the presence of 3 g. Raney Ni and
 treated with H at 75° and atmospheric pressure for 8 hrs. gave 2.2 g.
 3,3'-diamino-5,5',6,6',7,7',8,8'-octahydro-2,2'-binaphthyl (VIII), m.
 193-4° (EtOAc-alc.). Reduction at room temperature with N₂H₄ in the
 presence of Raney Ni or Pd-C was not successful. VIII (0.15 g.)
 in 1.5 cc. HCl and 2 cc. H₂O was treated at 0° with 0.2 g. NaNO₂ in
 1 cc. H₂O, the diazonium solution decomposed by addition to 0.3 g. iodine and
 1 g. KI in 20 cc. H₂O, after 1 hr. the solids collected, dried, and extracted with
 C₆H₆ to give 92% II, decompose 240-5°. The C₆H₆ extract washed with aqueous
 Na₂S₂O₃, dried, and chromatographed on Al₂O₃ gave 0.016 g. green crystals,

m. 208°. An intimate mixture of 0.3 g. II and 3 g. cuprous oxide under N at 0.3 mm. heated 0.5 hr. at 340°, the temperature kept 1 hr. at 220°, and the product extracted 8 hrs. in a Soxhlet apparatus gave yellow plates. The C6H6 mother liquor was chromatographed on Al2O3 giving a yellow solid. When freshly prepared Cu2O was used, 0.288 g. II afforded very thin plates of I, sublimed at 344-6° without melting. The 3 mg. yellow needles m. 224-6°. With an old sample of Cu2O, 0.3 g. II gave 4 mg. of product, m. 300-2°, considered to be a tetrahydrodibenzodiphenylene and 4 mg. of needles, m. 224-6°. The appropriate diamine (0.5 g.) in a solution of 0.4 g. NaNO2 and 4 cc. H2SO4 was added below 30° to 9 cc. AcOH, after stirring 1 hr. 100 cc. cold Et2O added, the mixture left 1 hr. at room temperature, the III collected, washed, dried, ground with 7 g. Cu2O, covered with 5 g. more Cu2O, and the whole pyrolyzed at dull red heat and 0.3 mm., cooled in vacuo, extracted with C6H6, and concentrated to give 5 mg. of a dibenzocarbazole, m. 299-301°. Chromatography of the mother liquor gave 36 mg. of an octahydrodibenzocarbazole, m. 157-8° (alc.), and the residue of 3 mg. was not further investigated. IV (11 g.) in 120 cc. Et2O added during 0.5 hr. to a mixture of 1.75 g. Mg and 50 cc. Et2O, the reaction being initiated by addition of iodine and a trace of MeMgI, and conducted under N. After refluxing 1 hr. 60 cc. dry C6H6 was added at 0° and the refluxing continued 1 hr. Unchanged Mg was removed and carboxylation carried out by pouring on to a slurry of Et2O and solid CO2 followed by acidification with 500 cc. 20% HCl; fractional crystallization gave 0.5 g. 2,2'-binaphthyl, 12 mg. of orange plates, m. 271° (red trinitrofluorenone derivative of m. 267°), and a heavy viscous liquid which could not be distilled at 3 mm.

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DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE	TOTAL

CA SUBSCRIBER PRICE ENTRY SESSION
-14.40 -14.40

=> d his

(FILE 'HOME' ENTERED AT 10:40:00 ON 18 SEP 2008)

FILE 'REGISTRY' ENTERED AT 10:40:21 ON 18 SEP 2008

L1 80 S (2-7)/LI AND P AND 3.5-8/O

FILE 'CAPLUS' ENTERED AT 10:40:52 ON 18 SEP 2008

L2 59 S L1

L3 8 S L2 AND (TI OR V OR CR OR MN)

L4 11 S L2 AND (FE OR CO OR NI OR CU OR ZR)

L5 6 S L2 AND (NB OR MO OR RU OR AG OR TA OR W OR PT OR AU)

L6 18 S L3 OR L4 OR L5

=> s l1

L7 59 L1

=> s l7 not nitride

276980 NITRIDE

34076 NITRIDES

285950 NITRIDE

(NITRIDE OR NITRIDES)

L8 55 L7 NOT NITRIDE

=> s l8 and (electrolyte or ion?)

278071 ELECTROLYTE

143681 ELECTROLYTES

333082 ELECTROLYTE

(ELECTROLYTE OR ELECTROLYTES)

2212647 ION?

L9 1 L8 AND (ELECTROLYTE OR ION?)

=> d

L9 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2008 ACS on STN

AN 2006:558613 CAPLUS <<LOGINID::20080918>>

DN 145:66266

TI Nonaqueous electrolyte secondary batteries having lithium phosphorus fluoride oxide or sodium/potassium hydrogen fluoride on surface or interface of cathode active mass particles

IN Kamibo, Yasushi

PA GS Yuasa Crop., Japan

SO Jpn. Kokai Tokkyo Koho, 15 pp.

CODEN: JKXXAF

DT Patent

LA Japanese

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	JP 2006156008	A	20060615	JP 2004-342240	20041126
PRAI	JP 2004-342240		20041126		

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L9 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 2006:558613 CAPLUS <<LOGINID::20080918>>

DOCUMENT NUMBER: 145:66266

TITLE: Nonaqueous electrolyte secondary batteries
 having lithium phosphorus fluoride oxide or
 sodium/potassium hydrogen fluoride on surface or
 interface of cathode active mass particles
 INVENTOR(S): Kamibo, Yasushi
 PATENT ASSIGNEE(S): GS Yuasa Crop., Japan
 SOURCE: Jpn. Kokai Tokkyo Koho, 15 pp.
 CODEN: JKXXAF
 DOCUMENT TYPE: Patent
 LANGUAGE: Japanese
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2006156008	A	20060615	JP 2004-342240	20041126

PRIORITY APPLN. INFO.: JP 2004-342240 20041126
 TI Nonaqueous electrolyte secondary batteries having lithium
 phosphorus fluoride oxide or sodium/potassium hydrogen fluoride on surface
 or interface of cathode active mass particles
 IT Secondary batteries
 (lithium; nonaq. electrolyte secondary batteries having
 lithium phosphorus fluoride oxide or sodium/potassium hydrogen fluoride
 on surface or interface of cathode active mass particles)
 IT Battery cathodes
 Safety
 (nonaq. electrolyte secondary batteries having lithium
 phosphorus fluoride oxide or sodium/potassium hydrogen fluoride on
 surface or interface of cathode active mass particles)
 IT 1333-83-1, Sodium fluoride (Na(HF₂)) 7789-29-9, Potassium hydrogen
 fluoride (KHF₂) 15772-79-9 845910-47-6, Lithium phosphorus fluoride
 oxide (LiPF₂O₂) 890852-51-4, Lithium phosphorus fluoride oxide
 (Li₂PF₃O₂) 890852-52-5, Lithium fluoride oxide phosphide
 (LiO-4F0-6O0-4P)
 RL: DEV (Device component use); USES (Uses)
 (nonaq. electrolyte secondary batteries having lithium
 phosphorus fluoride oxide or sodium/potassium hydrogen fluoride on
 surface or interface of cathode active mass particles)
 AB The batteries have, on surface and/or interface of cathode active mass
 particles, compds. containing alkali metals, F, and P, O, and/or H. The
 compds. prevent oxidative decomposition of electrolyte solvents on
 the surface of the active mass particles and improve safety of the
 batteries, especially when excess charging at high temperature

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